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<b>14. ABSTRACT</b>  Research efforts worldwide span diverse areas, including new materials such as nanotubes and nanofibers, miniaturization of electronics through nanotechnology, or integration of several sensors in artificial noses, to name only a few. Most of the time, the research results are reported, sometime as brief communications, at international conferences spread over different disciplines, such as materials science, electronic engineering, optics and photonics, nanotechnology, biochemistry and medicine, information technology, etc. It is therefore not very easy for young researchers to collect the most advanced background information. This Advanced Study Institute aims at providing young researchers with the most recent knowledge in sensing materials and sensor technologies, and with a unique opportunity to meet and discuss with outstanding specialists in these areas. The ASI will cover a wide range of materials and technologies encompassing semiconductors, self-assembled and biologically inspired nanomaterials, and photonic microsystems for the detection of various threats in applications spanning from environmental and health monitoring to security. Lectures on advanced theories and modeling of the sensing mechanisms will also be included.					
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Science for Peace and Security (SPS)  
North Atlantic Treaty Organisation

*NATO Advanced Study Institute*

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# ***Sensors for Environment, Health and Security: Advanced Materials and Technologies***

*VICHY (France), 16-27 September 2007*



CERAMEC

Centre Européen de Recherche sur les Applications Médicales et Environnementales des Céramiques  
European Research Centre for Environmental and Medical Applications of Ceramics



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Centre Européen de Recherche sur les Applications Médicales et Environnementales des Céramiques  
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European Research Center for Environmental and  
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Limoges (France)

# ***Practical Information***

## **Meeting Venue**

### ***Palais des Congrès-Opéra***

5 rue du Casino, Vichy

### **ASI entrance: Parc des Sources**

Registration, Poster exhibition, Breaks: “*Napoleon III*” hall

Lectures: “*Relais des Parcs*” room

Opening hours: 8:30am-12:00 and 1:30pm-6:00pm

### ***Aletti Palace Hotel***

3 place Joseph Aletti, Vichy

Tel: +33 (0) 470 30 2020

## **Meals**

Meals are served at the Aletti Palace Hotel

Breakfast: from 7:00am to 9:00am

Lunch: 12:30

Dinner: 7:30pm

Banquet (Thursday September 20) is served in the Berlioz ballroom of the Opéra at 8:00pm

## **Internet**

ISDN access available in the Conference Center (*Napoleon III* hall). No computer provided.

Wifi access available in the lobby of the Aletti Palace Hotel.

***Badges are mandatory for all ASI activities***

***Cell phones must be switched off during the sessions***

***Smoking is prohibited inside the conference center***

# Scientific Programme

## Sessions:

1. *Advanced Materials and Technologies*
2. *Sensors for Health*
3. *Sensors for Environment*
4. *Sensors for Security and Safety*

## Contributions:

- L: Plenary Lecture*  
*S: Seminar*  
*O: Oral contribution*  
*P: Poster*

*In the Book of Abstracts, the numbering follows the alphabetic order of the presenting author's name*

## Programme at a glance

<b>Sunday 16 September</b>	Registration Welcome party	<i>Napoléon III hall</i>
<b>Monday 17 September</b>	<i>Advanced Materials and Technologies</i>	<i>Relais des Parcs room</i>
<b>Tuesday 18 September</b>	<i>Sensors for Health</i>	<i>Relais des Parcs room</i>
<b>Wednesday 19 September</b>	<i>Advanced Materials and Technologies</i>	<i>Relais des Parcs room</i>
<b>Thursday 20 September</b>	<i>Sensors for Environment</i> Round table discussion on International Cooperation Banquet	<i>Relais des Parcs room</i> <i>Relais des Parcs room</i> <i>Opéra: Berlioz ballroom</i>
<b>Friday 21 September</b>	<i>Advanced Materials and Technologies</i> Poster session	<i>Relais des Parcs room</i> <i>Napoléon III hall</i>
<b>Saturday 22 September</b>	<i>Sensors for Health</i> Round table discussion on emerging markets and industrial needs	<i>Relais des Parcs room</i> <i>Relais des Parcs room</i>
<b>Sunday 23 September</b>	Excursion	
<b>Monday 24 September</b>	<i>Sensors for Security and Safety</i>	<i>Aletti Palace Hotel</i>
<b>Tuesday 25 September</b>	<i>Advanced Materials and Technologies</i>	<i>Aletti Palace Hotel</i>
<b>Wednesday 26 September</b>	<i>Sensors for Health</i>	<i>Aletti Palace Hotel</i>
<b>Thursday 27 September</b>	Closing session	<i>Aletti Palace Hotel</i>



## 1. Sunday 16 September

16:00 – 19:00		Registration
19:00 – 20:00		Welcome party
20:00		Buffet dinner

## 2. Monday 17 September

### *Session 1a: Advanced Materials and Technologies*

9:30 – 10:15		<b>Marie-Isabelle BARATON and Pavel KASHKAROV</b> Welcome address Introduction to the Advanced Study Institute
10:15 – 10:30		Welcome address by the Mayor of Vichy ( <i>to be confirmed</i> )
10:30 – 11:00		Break
11:00 – 12:00	L21	<b>Sudipta SEAL</b> Advanced nanomaterials for sensing
12:30		Lunch
14:10 – 15:10	L3	<b><u>Alexander GASKOV</u>, Marina RUMYANTSEVA and Andrey RYZHIKOV</b> Selective materials for gas sensor applications
15:10 – 15:30	O4	<b><u>Csaba BALÁZSI</u>, Lisheng WANG, Krithika KALYANASUNDARAM, Katarina SEDLACKOVA, Esra OZKAN ZAYIM, Imre Miklós SZILÁGYI, Judit PFEIFER, Attila L. TÓTH and Pelagia-Irene GOUMA</b> Synthesis and examination of hexagonal tungsten oxide nanocrystals for electrochromic and sensing applications
15:30 – 16:00		Break
16:00 – 17:00	L1	<b>Marie-Isabelle BARATON</b> Spectroscopic study of the gas detection mechanism by semiconductor chemical sensors
17:00 – 18:00	S5	<b>Florica MANEA, Dana PERNIU and <u>Joop SCHOONMAN</u></b> Defect chemistry of sensor materials
19:30		Dinner



### 3. Tuesday 18 September

#### *Session 2a: Sensors for Health*

9:20 – 10:20	L19	<b>Andrew MILLS</b> Optical sensors for carbon dioxide and their applications
10:20 – 10:50		Break
10:50 – 11:50	L7	<b><u>Sergei IGNATOV</u> and David WALT</b> Bacteria detection – Biosensors
12:30		Lunch
14:00 – 15:00	L9	<b>Arkady KARYAKIN</b> Electrochemical biosensors
15:00 – 16:00	L20	<b>Andrew MILLS</b> Oxygen indicators in food packaging
16:00 – 16:30		Break
16:30 – 16:50	O17	<b><u>Gayane MINASYAN</u>, Gagik AYVAZIAN and Artashes CHOMOYAN</b> Porous silicon bio-chemical sensors
16:50 – 17:10	O12	<b><u>Aisha HAYNES</u> and Perena GOUMA</b> Multi-functional nanomaterials for advanced biological/chemical sensor technologies
17:30		Tour of the Opera ( <i>to be confirmed</i> )
19:30		Dinner

#### 4. Wednesday 19 September

##### *Session 1b: Advanced Materials and Technologies*

9:00 – 10:00	L13	<b><i>G. KIRIAKIDIS and S. SADALE</i></b> Material growth and fundamental material characterization techniques
10:00 – 10:20	O18	<b><i>Aleksey VASILIEV, Roman PAVELKO, Vladimir SEVASTYANOV and Xavier VILANOVA</i></b> New approaches to synthesis of sensing materials for thermocatalytic and semiconducting gas sensors
10:20 – 10:50		Break
10:50 – 11:50	L11	<b><i>Pavel KASHKAROV</i></b> Sensing of donor and acceptor molecules in ensembles of silicon nanocrystals
12:30		Lunch
14:00 – 15:00	S1	<b><i>Astrid AKSNES</i></b> Photonic sensors for health and environment monitoring
15:00 – 16:00	L23	<b><i>Victor TIMOSHENKO</i></b> Light-induced generation of singlet oxygen in porous silicon
16:00 – 16:30		Break
16:30 – 16:50	O24	<b><i>Irina UVAROVA</i></b> Porous nanostructured ceramic sensors of humidity
18:00		Reception at the City Hall ( <i>to be confirmed</i> )
19:30		Dinner

## 5. Thursday 20 September

### Session 3: Sensors for Environment

9:00 – 10:00	L2	<b>Marie-Isabelle BARATON</b> Chemical sensors for outdoor air quality monitoring
10:00 – 10:20	O19	<b><u>Oleg M. POKROVSKY</u> and Jean-Louis ROUJEAN</b> The monitoring of the land surface biomass by means of the multi-angular sensors – A way to enhance the environmental and social security
10:20 – 10:50		Break
10:50 – 11:50	L25	<b>Ashok VASEASHTA</b> Nanomaterials in environmental pollution detection, monitoring, and remediation
11:50 – 12:10	O13	<b>Mustafa PETEK and <u>Bekir KARLIK</u></b> Determination of the mutagenic effects of pollution by AMES and neural networks
12:30		Lunch
14:00 – 15:00	L17	<b>Pedro MEDELIUS</b> Sensors for monitoring air quality in earth and space environment
15:00 – 15:20	O8	<b><u>J. A. GABALDÓN</u>, A. MARTINEZ, M.A. GONZALEZ-MARTINEZ, R. PUCHADES and A. MAQUIEIRA</b> Basic development of flow immunosensors for organic pollutants
15:20 – 15:40	O11	<b><u>Ana-Maria GURBAN</u>, Lucian ROTARIU, Madalina TUDORACHE and Camelia BALA</b> Development of biological sensors based on screen-printed electrodes for environmental pollution monitoring
15:40 – 16:00	O9	<b><u>George GALLIOS</u> and Miroslava VACLAVIKOVA</b> Magnetic iron oxides as possible sensors for toxic anions in water streams
16:00 – 16:30		Break
16:30 – 18:00		Round table discussion on International Cooperation: Talks from representatives of: EC, NIH, NSF, STCU <i>(to be confirmed)</i>
20:00 – 24:00		Banquet

## 6. Friday 21 September

### *Session 1c Advanced Materials and Technologies*

9:00 – 10:00	L4	<b><u>Alexander GASKOV</u> and Marina RUMYANTSEVA</b> Metal oxide nanocomposites: synthesis and characterization in relation with gas sensing phenomena
10:00 – 10:30	O23	<b><u>Martin HEBEL</u> and <u>Pier Andrea SERRA</u></b> Development of a parallel-computing embedded telemetry system for voltammetric microsensor and biosensor applications
10:30 – 11:00		Break
11:00 – 12:00	L22	<b><u>Sudipta SEAL</u></b> Nanosensors
12:30		Lunch
14:00 – 14:20	O2	<b><u>Teresa ANDREU</u>, <u>Luís FERNÁNDEZ</u>, <u>Jordi ARBIOL</u> and <u>Joan R. MORANTE</u></b> Mesoporous indium oxide for gas sensor applications
14:20 – 14:40	O14	<b><u>Ganna KHARLAMOVA</u>, <u>Natalia KIRILLOVA</u> and <u>Marina BONDARENKO</u></b> Novel molecular crystals of carbon as new perspective elements of nanosensors
14:40 – 15:00	O5	<b><u>Arezki BENFDILA</u></b> Metal oxide silicon based advanced sensors
15:00 – 15:20	O20	<b><u>Sameer DESHPANDE</u>, <u>David REID</u>, <u>Christina DRAKE</u>, <u>Peng ZHANG</u>, <u>Hyoung J. CHO</u> and <u>Sudipta SEAL</u></b> Nano-crystalline indium doped tin oxide gas sensor
15:20 – 16:00	P	Poster session
16:00 – 16:30		Break
16:30 – 18:00	P	Poster session
19:30		Dinner

## 7. Saturday 22 September

### *Session 2b: Sensors for Health*

9:00 – 10:00	L15	<b>Wim LAUREYN</b> Microelectronics based biosensors for the detection of proteins, nucleic acids and cells
10:00 – 10:30	O10	<b><i>I. CIMALLA, M. GEBINOVA, M. KLETT, V. LEBEDEV, K. TONISCH, V. CIMALLA, O. AMBACHER and A. SCHOB</i></b> Response of nerve cell to inhibitors recorded by AlGaIn/GaN field effect transistors
10:30 – 11:00		Break
11:00 – 12:00	L8	<b><i>Sergei IGNATOV, Alexander VOLOSHIN and Tatiana IGNATYUK</i></b> Bionano-Microbiology
12:30		Lunch
14:00 – 15:00	S2	<b>David M. BALSHAW</b> Sensor technologies for linking information on exposure, early biological response and disease progression
15:00 – 16:00	L24	<b>Victor TIMOSHENKO</b> Singlet oxygen generation and detection for biomedical applications
16:00 – 16:30		Break
16:30 – 18:00		Round table discussion on emerging markets and industrial needs (including a 30-minute talk) <i>(to be confirmed)</i>
19:30		Dinner

## 8. Sunday 23 September

<i>Departure time: to be announced</i>		Excursion ( <i>Programme to be announced</i> )
19:30		Dinner

## 9. Monday 24 September

### *Session 4: Sensors for Security and Safety*

9:00 – 10:00	L5	<b><u>James HARDY</u>, R. J. WARMACK and N. V. LAVRIK</b> Sensor science for national security
10:00 – 10:30	O16	<b><u>Show-Ling LEE-MÜLLER</u>, Gerd SCHUMACHER and Alan SMITH</b> A roadmap for materials for security
10:30 – 11:00		Break
11:00 – 12:00	L27	<b><u>Alexey VASILIEV</u>, Vladimir FILIPPOV, Alexander TERENCEV and Werner MORITZ</b> MIS-structure sensors with solid electrolyte gate layer for detection of explosive and hazardous gases
12:30		Lunch
14:00 – 15:00	L6	<b><u>James HARDY</u> and Michael WRIGHT</b> Radiation and nuclear detection research and development
15:00 – 16:00	L28	<b><u>Alexey VASILIEV</u>, Vittorio GUARNIERI, Mario ZEN, Leandro LORENZELLI, Sergey GOGISH-KLUSHIN, Dmitry KHARITONOV, Andrey SOKOLOV, Nikolay SAMOTAIEV and Roman PAVELKO</b> Sensors based on technology “nano-on-micro” for wireless instruments preventing ecological and industrial catastrophes
16:00 – 16:30		Break
16:30 – 17:30	S3	<b><u>Greg HORLER</u></b> Near field communications for sensing and security
17:30 – 17:50	O15	<b><u>Adam KRAUSE</u> and Thomas THUNDAT</b> Selective detection of trace explosives using piezoresistive microcantilevers
19:30		Dinner

## 10. Tuesday 25 September

### *Session 1d: Advanced Materials and Technologies*

9:00 – 10:00	L14	<b><u>G. KIRIAKIDIS</u> and S. SADALE</b> Systems and set-ups for effective sensing response applications
10:00 – 10:20	O3	<b><u>E. G. APUSHKINSKY</u> and <u>M. S. ASTROV</u></b> Correlation analysis of the noise sensor signals by the adaptive processor operating using a nuclear spin echo phenomenon
10:20 – 10:50		Break
10:50 – 11:50	S4	<b><u>Michael KRAFT</u> and Yufeng DONG</b> Sigma delta modulator interfaces for MEMS capacitive sensors
12:30		Lunch
14:00 – 15:00	L12	<b><u>Pavel KASHKAROV</u></b> Sensing of dielectric liquids in porous silicon matrix
15:00 – 15:20	O21	<b><u>Zenon SARBAK</u></b> Active centres for carbon monoxide chemisorption on Pt-Sn/Al <sub>2</sub> O <sub>3</sub>
15:40 – 16:00	O22	<b><u>Katarína SEDLÁČKOVÁ</u>, <u>Peter LOBOTKA</u>, <u>Ivo VÁVRA</u>, <u>T. LALINSKÝ</u>, <u>Š. CHROMÍK</u>, <u>M. ŠPANKOVÁ</u>, <u>J. DÉRER</u> and <u>V. ŠMATKO</u></b> LSMO microbolometer for detection of THz radiation
16:00– 16:30		Break
16:30 – 16:50	O1	<b><u>Sergey AIZIKOVICH</u> and <u>Elena AMBALOVA</u></b> The elastic half-plane with a thin inhomogeneous coating under the action of a stretching force applied at infinity
16:50 – 17:10	O7	<b><u>Janusz E. DMOCHOWSKI</u></b> Donors in semiconductors: The past or the future of semiconductor electronics ?



## 11. Wednesday 26 September

### *Session 2c: Sensors for Health*

11:00 – 12:00	L26	<b>Ashok VASEASHTA</b> Dynamics and transport of nanomaterials in the environment and human health implications: An ontological modality
12:30		Lunch
14:00 – 15:00	L16	<b>Wim LAUREYN</b> Surface chemistry to bridge inorganic biosensor surfaces and biological materials
15:00 – 16:00	L10	<b>Arkady A. KARYAKIN</b> Bioelectrocatalysis
16:00 – 16:30		Break
16:30 – 17:30	L18	<b>Pedro MEDELIUS</b> Sensory conversion devices
17:30 – 17:50	O6	<b><i>Ivan A. BOLSHAKOV, Nataliya A. VAVILOVA and Arkady A. KARYAKIN</i></b> Microsensors based on nanosized structures of Prussian blue for hydrogen peroxide detection
19:30		Dinner

## 12. Thursday 27 September

9:30 – 10:30		Closing session Feedback from participants
10:30 – 11:00		Coffee
Departure of participants		

## POSTERS

P1	<b><u>Sergey AIZIKOVICH</u>, Leonid KRENEV and Irina TRUBCHIK</b> The identification of the properties of graded and biologically inspired materials using nanoindentation
P2	<b><u>Astrid AKSNES</u>, Hanne MARTINUSSEN and Helge ENGAN</b> Smart microsystem for diagnostic imaging of vulnerable plaque
P3	<b><u>Katalin ALBRECHT</u> and Zsolt PUSKÁS</b> Measuring the protein binding capability of biocompatible surfaces with label-free biosensor
P4	<b><u>Burcu Selin AYTAZ</u>, Ufuk BAKIR and Gürkan KARAKAS</b> Preparation of ENFET type glucose biosensor
P5	<b><u>N.V. BOSHITSKAYA</u>, I.V. UVAROVA and H.A. IVASCHENKO</b> Carbon nanotubes as biosensors. Stability of carbon nanotubes in human blood plasma
P6	<b><u>Nuray DEMIREL</u> and Samuel FRIMPONG</b> Monitoring environmental impacts of surface mining using spot high resolution visible (HRV) sensor systems
P7	<b><u>Dilek DÜNDAR</u>, Mika HARBECK, Ilke GÜROL, E. MUSLUOĞLU, Z.Z. ÖZTÜRK and V. AHSEN</b> Phthalocyanine coated QCM sensors for monitoring pollutants in aqueous media
P8	<b><u>P.A. FORSH</u>, M.N. MARTYSHOV, V.Yu. TIMOSHENKO, and P.K. KASHKAROV</b> Influence of adsorption of active molecules on electrical transport in nanostructured porous silicon
P9	<b><u>N.R. HAKOBYAN</u></b> New roles for IFT in cell function
P10	<b><u>Iana KALININA</u>, Galina SHEVCHENKO and Elizabeth KORDYUM</b> Gravity-induced changes in cortical microtubules in Arabidopsis thaliana root cells
P11	<b><u>S. KRAEVSKIY</u>, E. DUBROVIN, T. IGNATYUK, A. VOLOSHIN, I. YAMINSKY and S. IGNATOV</b> Direct investigation of bacteriophages-bacteria interaction
P12	<b><u>Lyubov KUNYTSKA</u></b> Nanocomposites for active elements of humidity's sensors
P13	<b><u>Florica MANEA</u>, Ciprian RADOVAN, Aniela POP, Ioana CORB, Georgeta BURTICA, Plamen MALCHEV, Stephen PICKEN and Joop SCHOONMAN</b> Carbon composite electrodes applied for electrochemical sensors
P14	<b><u>G. KORTIDIS</u>, K. MOSCHOVIS and G. KIRIAKIDIS</b> InO <sub>x</sub> layered Surface Acoustic Wave (SAW) devices for gas sensing applications
P15	<b><u>G. OSPANOVA</u>, M. TLEBAYEV, E. KOLTSOVA and T. BAYZHUMANOV</b> Technology for obtaining carbon nanotubes

P16	<b><i>Viktor KRAKhmaLEV and Adkham PAIZIEV</i></b> Visualization and detection of chemical compounds in blood smears
P17	<b><i>Adkham PAIZIEV and Viktor KRAKhmaLEV</i></b> In vivo observation the super weak luminescence of single living cotton cell
P18	<b><i>J. FRAISSARD, M. PETRYK, S. LECLERC and D. CANET</i></b> Use of NMR spectroscopy and zeolite crystallite materials to study of benzene adsorption kinetics
P19	<b><i>N. SAMOTAIEV, A.A. VASILIEV, A.V. SOKOLOV and B.I. PODLEPETSKY</i></b> The analysis of catalytic activity of Pd-SnO <sub>2</sub> gas sensing materials material in oxidation reaction
P20	<b><i>Stanislav TRASHIN, Mikhail VAGIN</i></b> Electroanalysis of redox-inactive proteins by liquid film-modified electrodes
P21	<b><i>A.S. VORONTSOV, V.A. DEMIN, Yu.V. RYABCHIKOV, V.Yu. TIMOSHENKO and P.K. KASHKAROV</i></b> Optical spectroscopy of silicon nanocrystals for biomedical applications
P22	<b><i>Lisheng WANG, P.I. GOUMA, Alexandra TELEKI and S.E. PRATSINIS</i></b> Synthesis of pure and doped WO <sub>3</sub> nanoparticles by flame spray pyrolysis and their gas sensing properties



# ***BOOK OF ABSTRACTS***

## ***1. Lectures***



## SPECTROSCOPIC STUDY OF THE GAS DETECTION MECHANISM BY SEMICONDUCTOR CHEMICAL SENSORS

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Fourier transform infrared (FTIR) spectroscopy is a very relevant tool for the characterization of the surface composition and surface reactivity of ceramic nanoparticles provided a specific setup is attached to the spectrometer. Surface chemical groups which originate from the synthesis process and/or from contamination by environment, are identified by *in situ* FTIR analysis. Moreover, the stability of these surface groups can be checked by thermal desorption up to 500°C under dynamic vacuum, thanks to the infrared vacuum cell. Surface reactive sites are characterized through the adsorption of various selected organic molecules (probe-molecules).

Once the nanoparticle surface is characterized, the surface chemical reactions can be monitored *in situ* when a gaseous media is adsorbed on the nanoparticles. Gas adsorption may lead to reversible phenomena under adsorption/desorption cycles with no contamination of the nanoparticle surface. But, in different cases, gas adsorption may cause the transformation of the surface chemical groups and/or the formation of new surface entities, thus modifying the surface reactivity. Irreversible adsorption of selected molecules is a way to functionalize surfaces.

Besides, the electronic absorption is an intrinsic part of the optical absorption but has often been ignored in vibrational studies because it is broad and does not correspond to narrow bands with defined absorption maxima. Yet, it can be shown that, according to the classical Drude model, the infrared absorption due to the free carriers is directly related to the square of the wavelength and its magnitude is proportional to the number of free carriers. Our work has shown that FTIR spectroscopy can be used to follow the variations of the free carrier absorption of semiconductor nanoparticles. In other words, it becomes possible to monitor the variations of the electrical conductivity of semiconductor nanoparticles which are generated when the surrounding atmosphere is varied. In addition, we have proved that these variations of the electrical conductivity of nanoparticles monitored by FTIR spectroscopy can be directly related to the electrical response of the real sensors fabricated from the same nanoparticles.

We have applied this unusual feature offered by FTIR spectroscopy to the optimization of chemical gas sensors. Indeed, simultaneous study of the chemical reactions occurring at the nanoparticle surface under gas adsorption, on the one hand, and of the resulting variations of the electrical conductivity, on the other hand gives a wealth of information. This correlation of the surface reactions with the changes in the electrical conductivity is the fundamentals of the gas detection mechanism by semiconductor chemical sensors. From such experiments, it can be estimated whether the semiconductor nanoparticles are suitable for gas detection before embarking in the actual fabrication of the device.



**CHEMICAL SENSORS FOR OUTDOOR AIR QUALITY MONITORING****Marie-Isabelle BARATON***SPCTS – UMR CNRS, Faculty of Sciences, University of Limoges, FRANCE**E-mail: m-isabelle.baron@unilim.fr*

Air pollution has been one of Europe's main political concerns since the late 1970s. European Union policy on air quality aims to develop and implement appropriate instruments to improve air quality. As a result, the Member States have been instructed to establish a network of air quality monitoring (AQM) stations in their main cities and to inform citizens about the air quality on a daily basis. In the current AQM stations, the analyzers allow precise concentration measurements of different kinds of gaseous pollutants in air. But, they are expensive, complex and bulky equipment which do not allow "real time" dissemination of the information to the public because of the lengthy air sampling and data processing.

In a project (INTAIRNET) funded by the European Commission under the IST programme, our Consortium proposed alternative air quality monitoring microsystems based on cost-effective semiconductor gas sensors. This lecture will summarize our integrated approach to take up the challenge of definite improvement of the sensor sensitivity to detect very low levels of pollutants (CO, NO, NO<sub>2</sub>, O<sub>3</sub>) in air. Indeed, commercial semiconductor sensors do not meet the detection threshold criteria set by the official organizations in charge of environment protection. The performance of these commercial sensors have to be enhanced especially in terms of high sensitivity to gaseous pollutants and low cross-sensitivity to humidity. Our approach includes the use of metal oxide nanosized particles, the control of the size and surface chemistry of the nanoparticles, the optimization of the screen-printing process to the nanometer size specificity. All these necessary steps for sensor improvement will be discussed. It will be shown how, thanks to fundamental studies, it has been possible to transform bulky expensive AQM stations into cost-effective portable devices.

In the second stage of our concept, these portable gas sensing units associated with global positioning systems (GPS) communicate with a central computer via a wireless network based on the GSM protocol. These microstations, installed on mobile carriers such as city buses, would constitute a dynamic network covering the city and complementing the existing AQM stations. Through the Internet, it becomes therefore possible to not only inform in quasi real time citizens on air quality status but also help decision-makers to more efficiently manage road traffic and provide scientists with additional data to refine mathematical models of pollution clouds in cities.

It has to be emphasized that our proposed dynamic network, allowing rapid and versatile air quality monitoring in every city in every country at low cost, is relevant only if semiconductor sensors are successfully and reliably improved as presented in this lecture.

## SELECTIVE MATERIALS FOR GAS SENSOR APPLICATIONS

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The nanostructured semiconductor oxides: ZnO, SnO<sub>2</sub>, WO<sub>3</sub>, and In<sub>2</sub>O<sub>3</sub> are widely used as sensitive materials in metal oxide gas sensors (MOS). The advantages of semiconductor gas sensors: high sensitivity, fast response, small dimensions, low power consumption, and low cost have aroused the considerable interest to the application for air monitoring and in the different alarm systems. The main part of commercial gas sensors (Figaro, Drager, General Monitors) is based on tin oxide. SnO<sub>2</sub> surface exhibits good adsorption properties and reactivity due to free electron availability, presence of surface and bulk oxygen vacancies, and active chemisorbed oxygen (O<sub>2</sub><sup>-</sup>, O<sup>2-</sup>). Solid-gas interactions take place at moderated temperature through surface adsorption phenomena. The mechanism of electrical response of such materials is based on the reversible effect of band bending in the near surface layer caused by process of chemisorption. Depending on the atmosphere composition, a modification of the free electron density near the surface occurs and results in the conductivity change. The main problem of semiconductor sensors for the chemical threat agent detection is low selectivity, which gives rise to the probability of the false alarm. The limitation of selectivity is caused by non-selective character of adsorption process. The some approaches to improve the selectivity of MOS are discussed.

### Introduce the catalysts into semiconductor material

The recent work was focused on the development of new types of high selective sensor materials – complex oxide structure based on SnO<sub>2</sub> nanocrystallites coated with clusters of catalysts such as noble-like metals (Pt, Pd, Ru, Rh) or metal oxides (Fe<sub>2</sub>O<sub>3</sub>, CuO, NiO, CeO<sub>2</sub>, MoO<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, and La<sub>2</sub>O<sub>3</sub>) [1, 2]. These non-homogeneous nanostructured composite materials demonstrate higher thermal stability as compared with pure SnO<sub>2</sub> and exhibit the high selective sensitivity towards different reducing and oxidizing gases: H<sub>2</sub>, H<sub>2</sub>S, CH<sub>4</sub>, NH<sub>3</sub>, NO<sub>2</sub>, CO, and various organic products. The catalytic additives reduce interaction between the SnO<sub>2</sub> crystallites, inhibit the inter-diffusion of components, and therefore stabilize the structure and electrical properties of this material. In addition, the specificity of catalytic reactions allows the selective gas detection

### Development of catalytic filtering membrane.

Selectivity enhancement can be also achieved by selective processes of gas diffusion using two types of gas filters. Passive filtering membranes separate the gases as a function of size of gas molecules and their adsorption affinity to membrane material. Active (catalytic) membranes improve selectivity and stability of gas sensors also due to selective interactions with certain molecules. Various materials are used as filtering membranes in gas sensors: catalytic metals, porous dielectric oxides and zeolites, dielectric oxides doped with catalytic metals, pure and doped semiconductor oxides, double-layer structures of dielectric oxide and catalytic metal. The most used filters are pure and doped ceramic membranes on the surface of sensor element. The effect of ceramic membrane of Al<sub>2</sub>O<sub>3</sub>(M = Pd, Pt, Rh, Ru) deposited on the SnO<sub>2</sub>(Pd) thin films was discussed [3].

Thin film catalytic filtering membranes could improve significantly selectivity of semiconductor gas sensors. The membranes based on  $\text{Al}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3(\text{M})$ , where  $\text{M} = \text{Pd}, \text{Pt}, \text{Rh}, \text{Ru}$  of thickness 9-56 nm have been deposited on the surface of  $\text{SnO}_2(\text{Pd})$  thin films by aerosol pyrolysis method. Sensors properties of obtained  $\text{SnO}_2(\text{Pd})/\text{Al}_2\text{O}_3$  and  $\text{SnO}_2(\text{Pd})/\text{Al}_2\text{O}_3(\text{M})$  structures were tested under  $\text{H}_2$ ,  $\text{CO}$ ,  $\text{CH}_4$  and  $\text{C}_3\text{H}_8$ -air gas mixtures at  $100^\circ\text{--}300^\circ\text{C}$ . Sensitivity considerably depends on membrane thickness and doping metal. Membranes reduce significantly sensitivity to  $\text{CO}$  and  $\text{H}_2$ , but a selectivity of hydrogen detection in the presence of  $\text{CO}$  is improved. An increase of sensitivity to hydrocarbons at  $200^\circ\text{C}$  was observed. A selective detection of hydrocarbons in the presence of  $\text{CO}$  and  $\text{H}_2$  is shown. Selectivity enhancement by filtering membranes is probably related with complex processes of selective diffusion, adsorption and reactions on membrane surface. The possibility of selective detection of reducing gases was shown. The use of thin film catalytic filtering membranes deposited on the surface of sensing element is of high interest in multisensor systems for simultaneous selective detection of multiple gases.

#### The combination of Chemical sensor with Pre-concentrator.

The assembling of semiconductor materials with molecular sieves gives a new possibility to improve the selectivity of MOS (Fig.1). This concept has been validated by preliminary experiments and stimulated deep interest from industry officers. Among molecular sieve materials, microporous zeolites and mesoporous aluminosilicates are the most suitable candidates as pre-separators and pre-concentrators, because they exhibit very uniform pore distribution, which provides for the best sieving effects. The surface of these materials can be modified to ensure selective adsorption of required compounds. The recent achievements in the development of new micro/mesoporous composite materials with bimodal pore distribution open new horizons for the improvement of the selectivity of pre-separators and pre-concentrators due to the unique geometry of their pores and adjustable surface chemistry.

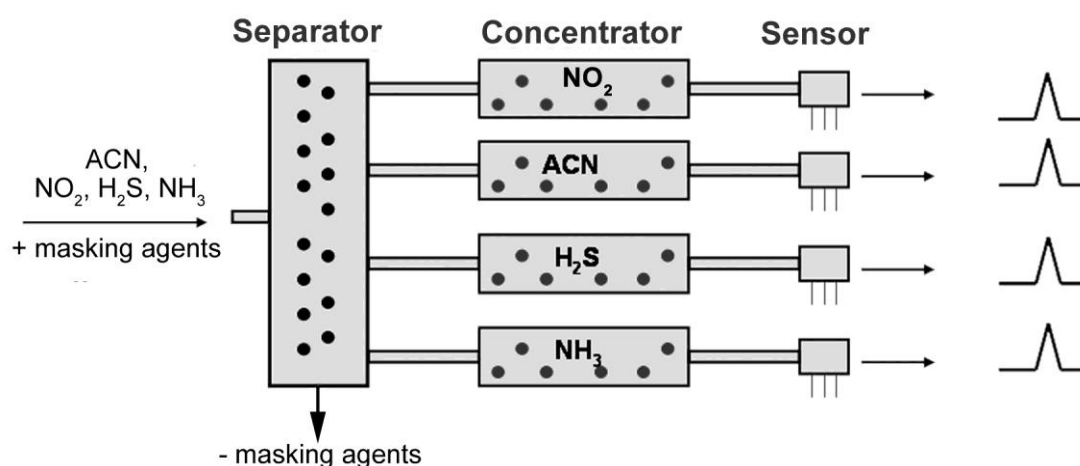


Fig.1. Scheme of “separator – concentrator – sensor” system.

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# METAL OXIDE NANOCOMPOSITES: SYNTHESIS AND CHARACTERIZATION IN RELATION WITH GAS SENSING PHENOMENA

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The novel class of advanced materials based on the nanometer-scaled heterogeneous metal oxides systems M1O-M2O (nanocomposites) is discussed regarding gas sensor applications. The materials are composed of nanocrystals of semiconductor oxides: M1O = SnO<sub>2</sub>, In<sub>2</sub>O<sub>3</sub>, WO<sub>3</sub> modified with catalysts: M2O = Fe<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, MoO<sub>3</sub>, WO<sub>3</sub>, CuO, NiO, CeO<sub>2</sub>, and La<sub>2</sub>O<sub>3</sub>. The selection of catalyst is destined to achieve the selectivity in the reactions of materials with specified gas molecules. The improving of selectivity could be reached either by increasing of oxidation/reduction activity or by control of concentration and nature of acid centers (Brønsted or Lewis type) on surface of semiconductor oxide [1-3].

The materials with grain size of 3-50 nm and specific surface area up to 200 m<sup>2</sup>g<sup>-1</sup> have been prepared as powders or thin films by chemical and physical routs: sol-gel, aerosol pyrolysis, laser deposition, and magnetron sputtering techniques. In the case of wet chemical deposition a co-precipitation and impregnation techniques with application of surfactant were used to obtain the samples. The variation of preparation conditions: nature of precursors, temperature of heat treatment gives rise to formation of materials of different crystallite size, depth and surface composition and diverse distribution of catalyst over the semiconductor oxide matrix. All these material characteristics impact on the gas sensor performance.

Application of different complementary techniques: X-ray diffraction, Transmission Electron Microscopy, Raman spectroscopy, Mössbauer spectroscopy, and BET measurements allowed to describe the crystal structure and mutual distribution of components in nanocomposites in the whole composition range. Acidic properties of the samples were characterized by NH<sub>3</sub>-TPD method. The gas sensor properties of materials were tested towards NH<sub>3</sub> and other reducing and oxidizing gases to compare the sensor signal with density and nature of acid centers controlled by catalyst.

The materials demonstrate the inhomogeneous structure. The most important effect obtained in mixing is the stabilization at very low values of the crystallite size, even after high temperature (700°C) thermal treatment as estimated from the peak broadening in XRD spectra. This has an importance for material stability in a possible use as gas sensors. The catalysts stabilize the microstructure of nanocrystalline semiconductor oxide M1O. For example in the nanocomposite (SnO<sub>2</sub>)<sub>1-x</sub>-(Fe<sub>2</sub>O<sub>3</sub>)<sub>x</sub> the lowest particle size and the narrowest distribution are observed for the Fe<sub>2</sub>O<sub>3</sub> content x=0.18 and the largest particle size are observed for pure Fe<sub>2</sub>O<sub>3</sub>.

Acidic properties of the nanocomposites are strongly depended on catalyst selected. For example the concentration of acid centers on the surface of SnO<sub>2</sub> increases with addition of acidic oxide MoO<sub>3</sub> and decreases with addition of basic oxide Fe<sub>2</sub>O<sub>3</sub> (Fig.1). The role of acid centers in sensor behavior of nanocomposites is complicated and depends on the mechanism

of the reactions of target molecules with active surface sites. In the case of  $\text{NH}_3$  detection when all types of surface-gas interaction take place with participation of acid centers the sensor signal value is in a good agreement with the total density of acid centers of Lewis and Brønsted types.

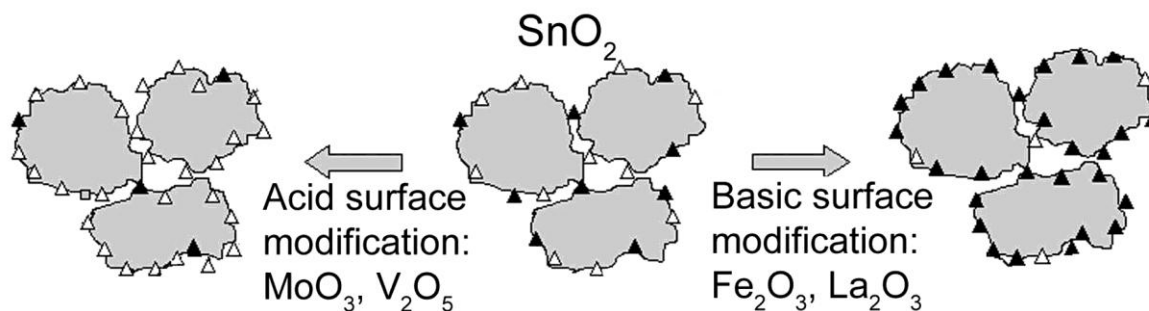


Fig.1. Surface chemistry of nanocomposites:  $\triangle$  – acid site,  $\blacktriangle$  – basic site.

For ethanol detection the sensor signal of materials mainly depends on the nature (Brønsted or Lewis type) of acid centers. There are two general ways of ethanol conversion: dehydration and oxidative dehydrogenation. The first one takes place mainly over the surface with Brønsted acidity and gives a low sensor response. On the contrary, dehydrogenation process needs Lewis acid-basic pairs and leads to high response due to reaction with chemisorbed oxygen  $\text{O}_s^-$ :



In metal oxide nanocomposites surface oxygen species with a negative charge and metal cations play a role of Lewis basic and acid centers respectively. Therefore, in the case of similar adsorptive ability of samples the selectivity of ethanol conversion into acetaldehyde and the sensor signal value depend on the ratio between strong (Lewis) and weak (Brønsted) acid centers. It means that over the surfaces showing mainly Brønsted acidity ethanol undergoes conversion via  $\text{C}_2\text{H}_4$  (dehydration) and such materials have poor sensitivity towards alcohol. On the contrary, samples with a dominant Lewis acidity show high response towards  $\text{C}_2\text{H}_5\text{OH}$  because of dehydrogenation conversion.

The work demonstrates the relationships between  $\text{SnO}_2$  sensor properties and density of acid centers. The increase of annealing temperature gives rise to the growth of part of strong acid centers for undoped  $\text{SnO}_2$ . The concentration of acid centers on the surface of  $\text{SnO}_2$  increases with addition of acidic oxide  $\text{MoO}_3$  and decreases with addition of basic oxide  $\text{Fe}_2\text{O}_3$ . Sensor signal towards  $\text{NH}_3$  correlates with the density of acid centers for both  $\text{SnO}_2\text{-Fe}_2\text{O}_3$  and  $\text{SnO}_2\text{-MoO}_3$  series. For ethanol detection the sensor signal mainly depends on the nature of acid centers (Brønsted or Lewis type).

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**SENSOR SCIENCE FOR NATIONAL SECURITY****James E. HARDY<sup>1\*</sup>, R. J. WARMACK<sup>1</sup> and N. V. LAVRIK<sup>1</sup>**<sup>1</sup> *Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA**\* Contact author: hardyje@ornl.gov*

Oak Ridge National Laboratory (ORNL) has over 200 professionals engaged in measurement science research and development. This work includes transduction techniques, advanced microelectronics, signal and image processing, modeling and simulation, material synthesis and characterization, and system engineering issues for packaging, miniaturization, integration, and communications (e.g. wireless networks). ORNL is applying this expertise to detecting, preventing, and reversing the proliferation of weapons of mass destruction, deploying integrated systems for incident awareness, detection, and response, providing technology for detection of explosives, and delivering enhanced protection and new capabilities to first responders and war fighters. Sensor systems include micro- and nano-technologies, optical, infrared (IR), and microwaves. Electronics' research has focused on low-power, low-cost, and wireless functionality and signal processing efforts cover data flow and conversion to information and knowledge for intelligent decision making.

The micro technology (micro-electromechanical systems - MEMS) has focused on arrays of microsensors to detect chemicals and biological items and IR imaging cameras. The microsensors designs include microcantilevers and microcapacitors in array format; typically comprised of 10 or more sensors. For chemical detection, pattern recognition techniques have been used to increase the selectivity of the sensor array. Much of the research has focused on coatings that will be sensitive to the analyte of interest and that will remain stable in a variety of environments. The microsensor array technology has been incorporated into a personal safety monitor for first responders and has been conceptualize for end-of-life detection in gas mask filters. Microcantilever arrays have been fabricated to form a low-cost, uncooled IR imaging camera with high sensitivity. Temperature differences of less than one degree have been detected. Nanotechnology has been used in developing better understanding at the cellular level and in medical research of transplants and skin grafts.

Significant research has been directed toward developing ultra low power electronics for signal conditioning and read out circuits for sensors and for RF circuits in wireless applications (RFID). Mixed signal (analog and digital) electronics has been developed to condition, filter, and communicate sensor data using only microwatts of power. Power management schemes have been created to optimize power consumption and select the best available power source (solar, batteries, line power, or scavenging from the environment). Innovations in signal and image processing have supported national security by helping improve quiet submarine technology and advancing image enhancement, face recognition, and scene/event analysis. The image analysis techniques have also made significant impacts in industry and in the medical field.

Major instrumentation systems have been developed and commercialized to rapidly detect chemical and biological warfare agents as well as persons hiding in vehicles by detecting their heart beat. Numerous optical-based systems have been created to monitor high valued assets and protect perimeters of critical infrastructures. Wireless systems have been developed to help achieve total asset visibility with the use of RFID tags, wireless communications, ad hoc networks, and software integration.

## **RADIATION AND NUCLEAR DETECTION RESEARCH AND DEVELOPMENT**

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Research and development is underway to improve radiation and nuclear detection capabilities. This research and development in radiation and nuclear detection includes areas such as advanced materials, applied research and engineering for designing and fabricating customized detection equipment, and theoretical, modeling and computational support. Oak Ridge National Laboratory (ORNL) has a distinctive set of detector materials fabrication and characterization capabilities and recently created a Center for Radiation Detection Materials and Systems. Applied research and engineering efforts have led to the development of improved detectors for specific applications including safeguards, treaty monitoring, and science experiments. All sizes, types, and capabilities of detector systems have been addressed from miniature to man-portable and from neutrons to gamma radiation. Dedicated test beds, in-house and in the field, have been established to analyze, characterize, and improve detection systems.

In addition to a complete set of materials fabrication capabilities, ORNL has some unique technologies such as radioactive crystal growth facilities and molecular jet chemical vapor deposition (patented by ORNL). ORNL also has comprehensive semiconductor and scintillator characterization tools including the highest flux neutron activation source for analysis of material purity and a diverse set of electronic, photonic, and radiation-base capabilities.

ORNL has develop a versatile and widely used nuclear measurement identification system (NMIS) that contains innovative signal processing and analysis technology to detect and measure highly enriched uranium (HEU). The system has application in non-proliferation activities and in monitoring conversion of HEU to reactor grade low enriched uranium (LEU). Over 10,000 neutron interrogation measurements have been performed with this NMIS. This system can also be used for imaging.

ORNL has a long legacy of experience with unique capabilities and facilities that has led to innovative detectors and analysis systems. The research portfolio ranges from basic material development to improving detector performance through fabricating, testing, optimizing, and deploying custom detector and systems.



## BACTERIA DETECTION - BIOSENSORS

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Biosensors promise to provide analytically powerful and inexpensive alternatives to standard bacteriological methods used today for bacteria detection. Biosensors are analytical devices that respond selectively to analytes or bacteria by use of a combination of a biological recognition system and a physical transducing element. The biological response is converted by the transducer to an electrical or optical signal that is directly related to the concentration of the analyte of interest or bacteria. A biosensor's recognition system may consist of one of many biological components including: enzymes, antigens, antibodies, nucleic acids, cells, cell organelles or receptor molecules. All of these biological components are capable of interacting very specifically with an analyte of interest or bacteria and to give a biosensor its unique specificity. The new biological recognition systems are very attractive for biosensoric bacterial detection method.

It is widely known that bacteria can be detected and distinguished by utilizing the unique recognition of the bacteria by specific bacteriophage strains. A number approaches have been developed to eliminate the time of specific bacteria/phage interaction.

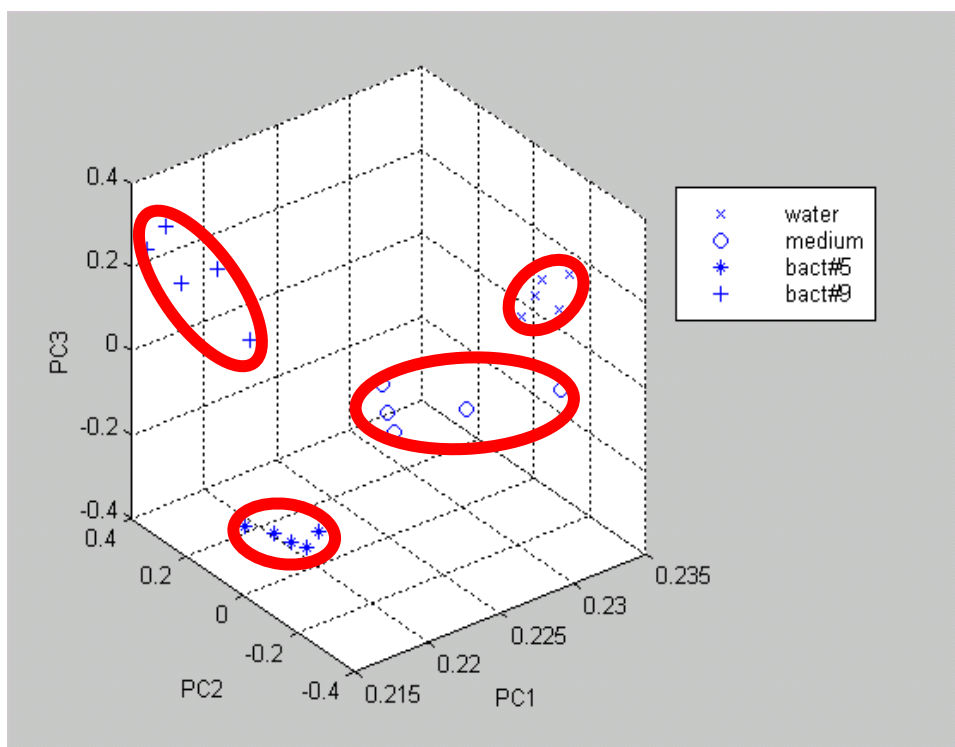
However, such methods have a number of limitations including loss of sensitivity and specificity of the phage/host interaction. Other methods of detection, which use native phage strains, rely on the formation of plaques in bacterial lawns. Such methods are lengthy to perform, requiring approximately 24 hours for a positive result, and have relatively low sensitivity.

In our Institute we use phages as a new recognition system for biosensor construction. The main idea of this project is the specific and partial disruption of the cell envelope of foodborne pathogen bacterial cells by phages and the registration of those injuries by adding metabolic substrates or dyes which are not penetrate to native bacterial cells. Adding of metabolite substrate or dyes to specific disrupted cells could induce oxygen consumption or reduction of dyes which are simple to detect by biosensor.

Another direction is devoted to the use of up-to-date remote method for bacteria detection with using an artificial nose system based on high-density optical biosensor arrays for the real-time microbes detection. This method will allow to detect bacteria without contact with biological material and will be very important in a wide range of disciplines, including medical analysis, food and environment.

The results of the investigations will enable us to open the new field of the bacteriology – aroma microbiology, and how smell of biological sample is correlated with the habitation environment and specific features of bacterial composition. They will provide us with a real opportunity to develop and apply to practice specific technologies associated with application

of fiber-optic biosensor to rapid and sensitive analysis of bacterial world. In this picture you can see preliminary results for non-contact bacteria identification with using artificial nose system based on fiber-optic sensor.



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## BIONANO-MICROBIOLOGY

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Nano-bio is a scientific area that integrates nano and bio to the benefit both. During the past decades, much progress has been made in the tool of nano-Atomic Force Microscopy (AFM). AFM belongs to a family of proximal probe microscopy techniques used for probing surface topography and properties on the atomic-molecular scale. The AFM microscopy provides 3-D images of the surface ultrastructures with molecular resolution, in real time and under physiological conditions by analysis interaction between surfaces and special tip named cantilever. The unique ability of AFM to image bacterial world at subnanometer resolution and in aqueous solution was opened new fields to investigate bacteria themselves and their surfaces under physiological conditions without any type of labeling. Applying AFM to analysis of bacteria requires a robust techniques for cell immobilization. Effective immobilization techniques must position the cells such that they are stable to tip forces and must be applicable to arrange of cell types. Strategies involving poly-L-lysine or gelatin modification of surface for immobilization bacterial cells or using cross-linking carboxyl groups on surface for fixation ammine groups located on bacterial surface has been presented. AFM can be used to distinguish various bacterial strains on the basis of their size and surface morphology. But in this case it is difficult to analyse images for identification of bacteria, especially in the presence of contaminants. To increase the sensitivity of immobilization of bacteria antibodies are used usually. Assays based on antibody-antigen binding are commonly used for bacterial identification due to the highly selective molecular recognition afforded by immune reaction between bacterial cells and specific antibodies. Present immobilization methods for antibodies are mainly based on silanized layer, polymer membrane, Langmuir-Blodgett film and self-assembled monolayer. AFM is used for the highly specific analysis of antibody-antigen binding. Conventional immunoassay techniques such as radioimmunoassay, enzyme immunoassay and surface plasmon resonance immunoassay are non-direct and based on statistical information. The ability AFM to image biological systems at high resolution makes AFM-immuno methods very attractive for direct analysis. For visualization antibody-antigen interaction gold colloidal particles were used. Immunoreagents were covalently attached to two-component self-assembled monolayers and characterized by AFM. Scanning microscopy was applied to the immunoassay of toxic bacterial protein by using self-assembling antibody layer on a protein A-coated surface. Sensitivity of AFM detection is very high and it is possible to detect of single antibody-antigen recognition events by force signal. In the case of bacteria, typical dimensions of bacterial cells (1-5µm) are suitable for AFM visualization without any labels. For the bacterial detection cantilever was modified by antibacterial antibodies and antibodies-bacteria interaction were estimated by changing of frequency parameters of cantilever.

In our Institute, we attempt to identify bacteria and bacterial spores of with using AFM. For specific bacterial visualization the new approach based on immobilization of specific

antibodies indirectly to the solid surface via antibody-binding staphylococcal protein A was developed. Protein A is known to capture specifically the Fc fragments of antibodies. Bacterial cells and spores have been chosen to discriminate objects on AFM pictures by forms and sizes in the presence of contaminants. The layer of antibodies against bacteria was exposed with bacteria and was used to evaluate the specificity with spores. The layer of antibodies against spores was exposed with spores and bacteria too. The AFM pictures were analyzed in the presence of bacterial cells or spores. We demonstrated the possibility of identification of bacteria by AFM with using antibodies immobilized to the surface through a protein A layer.

Moreover, we have elaborated the protocol of specimen preparation for visualization of interaction of bacteria with bacteriophages. For this purpose, solutions containing living bacterial cells *E.coli* and bacteriophages were mixed and placed in a thermostat at 37°C. In certain periods of time 5-10 µl of the mixture were taken from the solution in the thermostat for deposition onto mica and AFM monitoring. Preliminary results have shown the efficiency of AFM usage for these kinds of systems (Fig.1 - 2).

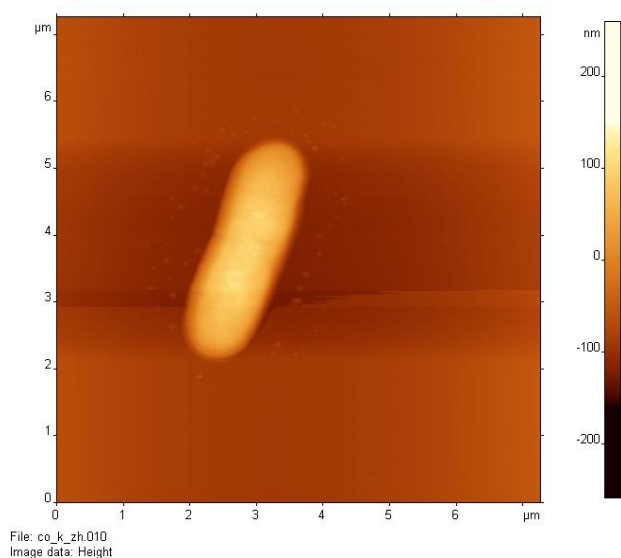


Fig. 1. AFM images of *E.coli* cells without phages

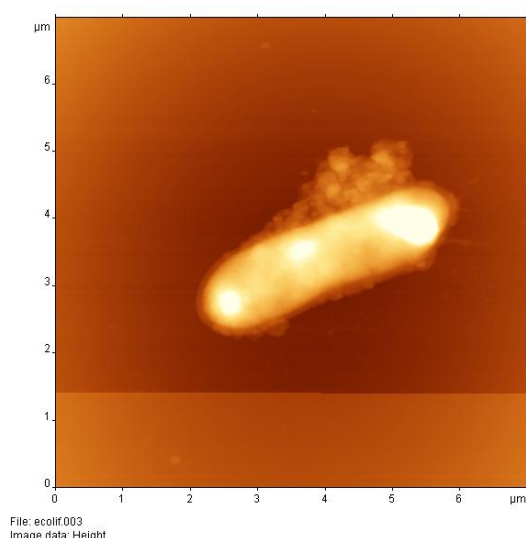


Fig. 2. AFM images of *E.coli* cells with phages. (35 min)

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**ELECTROCHEMICAL BIOSENSORS****Arkady A. KARYAKIN**

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First part of the lecture will be devoted to main definitions of biosensors. According to IUPAC, the biosensor consists of a transducer and a biological recognition element immobilized on it. To specify this broad definition, the additional requirements will be formulated. The scheme of biosensor operation will be presented and discussed.

A history of biosensor science will be outlined. The first successful publications on biosensors in 60s of XX century will be presented.

Classifications of biosensors will be presented. Specifically, biosensors can be classified according to the type of transducer and biorecognition mode. The examples of each type of biosensors will be presented. The advantages and limits of each type of biosensor will be discussed.

Electrochemical biosensors are divided into first, second, and third generation ones according to the principle for coupling of electrochemical and biochemical reactions. The first generation devices are based on detection of a coupled substrate or product of enzyme reaction. The second generation enzyme electrodes use mediator to deliver electrons between the enzyme active site and the electrode. The third generation biosensors use the phenomenon of direct bioelectrocatalysis.

Conducting polymers and their applications for biosensors will be presented. Special attention will be given to polyaniline capable for advanced potentiometric pH sensing.

Since more, than 90% of commercial biosensors are based on enzymes oxidases, the catalysis of the latter will be discussed. Special attention will be given to principles, how to elaborate electrochemical biosensors on the basis of oxidases.

Examples of the most successful biosensors, which revolutionized not only biosensor market, but also pharmaceutical market will be presented.

**BIOELECTROCATALYSIS****Arkady A. KARYAKIN**

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Bioelectrocatalysis is an acceleration of electrode reactions by biological catalysts. The latter are represented by the enzymes and the intact cells. The reasons for use of each type of biocatalyst and the general applications of bioelectrocatalysis will be discussed.

First part will be devoted to enzyme bioelectrocatalysis. The approaches, how to involve enzymes in bioelectrocatalysis, and the formulation of the direct bioelectrocatalysis will be presented. Thermodynamics of oxidase catalysis will be discussed. The two mediator based protocols for bioelectrocatalysis by the enzymes oxidases, which have found the most important practical applications, will be outlined. Special attention will be paid to dehydrogenase catalysis, which use  $\text{NAD(P)}^+|\text{NAD(P)H}$  redox couple as the natural electron mediator. Electrochemical regeneration of this particular redox mediator allows elaboration of biosensors for a great number of metabolites and fuel electrodes for variety of fuels.

The history of direct bioelectrocatalysis, starting from electrochemistry of redox enzymes will be presented. The most successful examples for the enzymes in direct bioelectrocatalysis (including information on the structure of the enzymes) will be outlined. The approaches, how to involve enzymes in direct bioelectrocatalysis and the fundamentals of this phenomenon will be discussed.

A rather novel phenomenon, the direct bioelectrocatalysis by intact cells, will be presented. An evidence for electroactivity of intact cells will be outlined, and the pictures, how bacteria are using electrode as an electron acceptor will be shown.

In conclusion, both advantages and disadvantages of bioelectrocatalysis will be discussed.

## SENSING OF DONOR AND ACCEPTOR MOLECULES IN ENSEMBLES OF SILICON NANOCRYSTALS

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Porous silicon (PSi) prepared by electrochemical etching of crystalline Si in hydrofluoric acid solution consists of Si nanocrystals with mean sizes of 1-10 nm, depending on the preparation conditions. PSi is an bright example of Si nanocrystal ensemble with huge specific surface area (up to  $10^3$  m<sup>2</sup>/g), which is open for the interaction with ambient molecules. We present an overview of the adsorption effect of donor-like (NH<sub>3</sub>, C<sub>5</sub>H<sub>5</sub>N, H<sub>2</sub>O, etc.) and acceptor-like (NO<sub>2</sub>, I<sub>2</sub>, O<sub>2</sub>, etc.) molecules on the electron and optical properties of PSi. The Fourier-transform infrared spectroscopy (FTIR) and electron paramagnetic resonance (EPR) methods are employed to investigate the free charge carrier concentration ( $N_p$ ,  $N_e$ ) and adsorption-induced surface modifications of Si nanocrystals.

PSi samples were prepared by electrochemical treatment of heavily doped *p*- and *n*-type Si wafers of the (100) surface orientation in a solution of HF(48%):C<sub>2</sub>H<sub>5</sub>OH=1:1. Obtained PSi films were separated from the substrates by a sharp increase of the etching current density. The free carrier concentration was estimated from the FTIR absorption spectra (see for details Ref.[1]). An EPR spectrometer (frequency 9.5 GHz, sensitivity  $5 \cdot 10^{10}$  spin/G) was employed to measure the defect density in PSi. The experiments were carried out at room temperature.

Figure 1 shows typical spectra of the absorption coefficient of meso-PSi film, formed from  $p^{++}$ -Si:B ( $N_0 \sim 10^{20}$  cm<sup>-3</sup>). The spectrum of as-prepared film consists of the absorption bands of different surface vibrations as Si-H<sub>x</sub> deformation modes, with the peak located at 660 cm<sup>-1</sup>, Si-H<sub>2</sub>-scissors vibrations at 910 cm<sup>-1</sup>, and Si-H<sub>x</sub>( $x=1,2,3$ ) stretching modes at 2070-2170 cm<sup>-1</sup>. The presence of these absorption bands evidences the hydrogen termination of PSi surface. Also the absorption spectrum exhibits a monotonically growing background with decreasing wavenumber, which is accounted for the free charge carrier absorption [5].

It was found that adsorption of acceptor-like molecules of NO<sub>2</sub> leads to a strong increase of the absorption coefficient of PSi due to the enhanced free charge carrier absorption. This effect is explained by the formation of acceptor-like species on Si nanocrystal surfaces in PSi films. Such complexes can be considered as acceptor dopants,

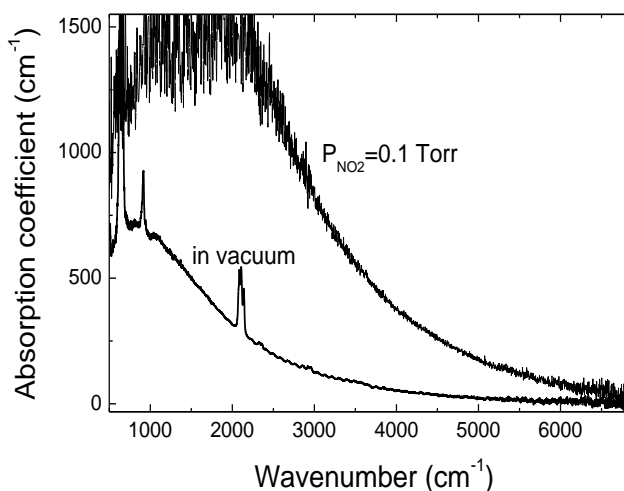


Fig. 1. Absorption spectra of as-prepared  $p^{++}$ -PSi films in vacuum or in NO<sub>2</sub> atmosphere.



which induce corresponding states in the forbidden gap of Si nanocrystals [1,2]. The adsorption-induced states are probably deep and hence they have high activation energies. However, the Coulomb interaction between adsorbed NO<sub>2</sub> molecules and surface defects ( $P_b$ -like centers), being possible due to small sizes of Si nanocrystals, leads to the appearance of donor–acceptor pairs as  $P_b^+-(NO_2)^-$ . The formation of these pairs results in the growth of  $N_p$  because of “passivation” of  $P_b$  centers due to their positive charging [8]. The above-proposed model is able to explain why the maximal  $N_p$  value after NO<sub>2</sub> adsorption does not exceed the substrate doping level.

Similar changes of the carrier concentration were obtained in our experiments with other molecules-strong acceptors of electrons. In case of I<sub>2</sub> molecules, the adsorption-induced changes of  $N_p$  were nearly reversible in adsorption/desorption circles at pressures up to the saturation one. The adsorption of donor molecules was found to result in a decrease  $N_p$  in  $p$ -

type PSi, while a growth of the free electrons was observed in  $n$ -type PSi. For example, as shown in Fig.2, the free electron concentration,  $N_e \sim 3 \cdot 10^{18} \text{ cm}^{-3}$  is achieved in  $n$ -type PSi after ammonia adsorption. The carrier concentration in  $p$ -type PSi exhibits a non-monotonic dependence on ammonia pressure, which indicates the transition from  $p$ - to  $n$ -type of the major carriers ( $N_p/N_e$  transition). The results obtained give evidences of the ammonia-induced states of shallow donors, which, together with the initial doping impurities and surface defects, determine the type and concentration of charge carriers in Si nanocrystals. Adsorption-related restructuring of Si nanocrystal surface bonds is confirmed by the EPR method, which detects new Si dangling bonds after NH<sub>3</sub> adsorption. The adsorption-induced  $P_b$ -like defects act also as traps for free charge carriers, which limit the carrier concentration at high pressure of ammonia similarly with the case of acceptor-like molecules of NO<sub>2</sub>.

Thus, the adsorption of active donor- or acceptor-like molecules can change the concentration of free charge carriers in meso-PSi from  $10^{17}$  to  $10^{19} \text{ cm}^{-3}$ . The experimental results are explained by a model, which takes into account the charging/recharging of the initial doping impurities and defects as well as the formation of new adsorption-induced states on Si nanocrystal surfaces.

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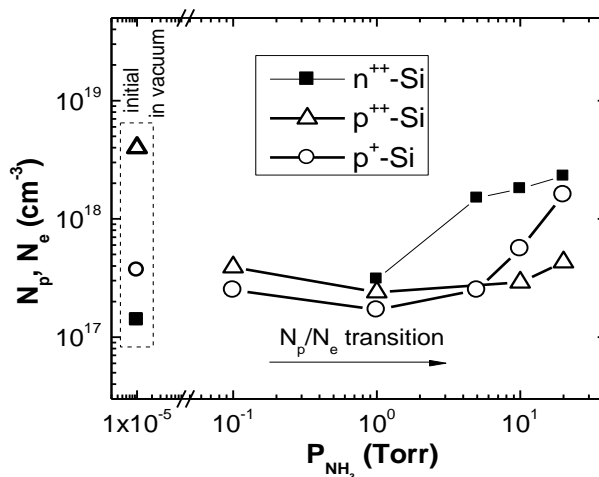


Fig. 2. Dependence of the free carrier concentration in meso-PSi films of different initial conductivity type and doping level vs NH<sub>3</sub> pressure.

**SENSING OF DIELECTRIC LIQUIDS IN POROUS SILICON MATRIX****Pavel KASHKAROV***Moscow State M.V. Lomonosov University, Physics Department, 119992 Moscow**RUSSIAN FEDERATION**E-mail: pavel@vega.phys.msu.ru; Tel: (+7)495 939 2193*

Porous silicon (PSi) obtained by an electrochemical etching of Si wafers exhibits unique electronic and optical properties, which are determined by the quantum confinement effect for charge carriers in residual Si material (nanocrystals as nanowires and quantum dots) and by the contrast between the dielectric functions of Si nanocrystals and pores (dielectric confinement) [1,2]. These properties of PSi enable us to recognize small concentrations of capillary-condensed dielectric liquids even with low refractive indices [3]. Also, the molecule adsorption/condensation can be monitored with fast optical response, which makes the PSi matrix a good candidate for sensor applications. We analyze the molecule adsorption and capillary condensation for organic (ethanol, acetone, benzene, etc.) dielectric substances in PSi nanopores with diameters of 1-10 nm. The appearance of the dielectric liquids in the nanopores are monitored by using measurements of the free-carrier absorption (FCA) in the infra-red (IR) spectral range and photoluminescence (PL) of excitons in Si quantum wires of PSi [4,5]. Theoretical analysis of the experimental data is based on a model, which takes into account both excitons and free charge carriers in Si nanowires surrounded by dielectric ambient [5].

It was found that the transients of FCA and PL of PSi showed similar decays with lifetime ranged from several to tens microsecond depending on temperature and dielectric properties of material embedded in the pores. Simultaneous decrease of the decay time of FCA and PL with increasing temperature from 100 to 300 K implies a strong coupling between subsystems of free carriers and excitons in the interconnected Si nanowires in PSi. The intensity of PL decreases and the amplitude of FCA increases when the nanopores are filled by media with the dielectric constant larger than that of silicon. This fact indicates a redistribution of carriers between the subsystems of free carriers and excitons. The temperature and dielectric dependent FCA and PL are well described by rate equations for excitons and free carriers, whose parameters (binding energy of excitons, lifetimes of radiative excitonic recombination and nonradiative free carrier recombination) are dependent on the dielectric properties of materials embedded in nanopores. According to the model, the exciton binding energy is controlled by the effective dielectric constant of PSi [5]. This leads to a drop of the exciton concentration and to a rise of the free carrier density when the effective dielectric function increases because of the filling of nanopores by dielectric liquids. Thus, the theoretical predictions are in agreement with the experimental observations. It opens possibilities for applications of PSi in sensors for dielectric liquids.

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## MATERIAL GROWTH AND FUNDAMENTAL MATERIAL CHARACTERIZATION TECHNIQUES

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Progress of science and technology depends profoundly on the proper choice of routes by which new substances/ materials are processed and their emerging properties. It is necessary to understand and control the macroscopic properties and functions of a material from a microscopic interpretation based on elementary constituents such as molecules, atoms, ions, and electrons, with their bonds and associated structures. The synthesis of materials so as to exhibit desired properties in light of a particular application is of immense importance.

In this presentation we are focusing on the various methods for the synthesis of transition metal oxides (TMOs) and their characterization by different methods targeting their gas sensing application. The most commonly used TMOs for gas sensing are ZnO, In<sub>2</sub>O<sub>3</sub>, WO<sub>3</sub>, SnO<sub>2</sub>. Amongst the various physical and chemical growth methods used for the growth of these materials, we will deal with pulse laser deposition (PLD), sputtering (dc and rf magnetron), aqueous chemical growth and spray pyrolysis will be discussed in more details. The more widely used fundamental structural and surface characterization techniques like X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), cross-sectional transmission electron microscopy (XTEM) and atomic force microscopy (AFM) will be reviewed.

The presentation of fabrication methods for these most commonly used metal oxides will be followed by a study on how growth techniques lead to nanostructures and nanostructured polycrystalline films with surface features of nanometer scale for film thickness below 1 μm. The presentation will continue with a discussion on how, a broad range of morphological parameters, affect the thin film response to various gases. After an overview, the study will focus on thin films prepared by the above techniques in different growth conditions. Metal oxides gas sensing properties particularly for In<sub>2</sub>O<sub>3</sub> and ZnO nanostructures and nanostructured thin films will be reviewed. It will be shown how the nanostructure and nanostructured surfaces are highly controlled by the deposition parameters, which, control the transport properties, and thus the sensing characteristics as measured by conductometric and Surface Acoustic Waves (SAWs) techniques.

After an overview, the discussion will focus on In<sub>2</sub>O<sub>3</sub> and ZnO thin films prepared for ozone sensing as they may exhibit resistivity changes of five to eight orders of magnitude at room temperature after exposure to UV light and subsequent ozone treatment and thus are candidates for low cost low operation temperature gas sensor applications.

As the microstructure, morphology and electronic structure of the metal oxides forms the basis for the metal oxides to exhibit different properties that in turn can be used for a particular application it is of utmost importance to understand the extent the desired properties are governed by the actual structural properties of the materials.

## SYSTEMS AND SET-UPS FOR EFFECTIVE SENSING RESPONSE APPLICATIONS

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A review of existing sensing response systems based on conductometric and SAW will be made. Proposals and designs for homemade lab prototypes from mask-making to gas manifolds and measurement systems will be accounted.

Some of the advantages of metal oxides sensors (MOS) are, a) their high sensitivity to most combustible gases including saturated hydrocarbons, NO and CO, b) their fast response time and reliability (simple set-up), c) their good resistance to corrosive gases and humidity, d) good mechanical strength and photo-plastic behaviour, and e) the low production cost. On the opposite side disadvantageous are their relatively poor selectivity, which, to some extent, may be improved by dopants and temperature adjustment during the measurement, and their relatively high power consuming operation temperature. Due to all these, one of the goals for the sensor technology is the production of low temperature working sensing systems.

The most commonly used techniques are based on conductometric systems. However, encouraging results are anticipated by current improvements by deployment of Surface Acoustic Wave sensors (SAWs). SAWs are becoming now a days some of the most commonly used sensor types in sensor array systems. The selectivity and sensitivity towards a certain analyte is achieved through coatings with different sensing layers chosen according to their strength of interaction with the analyte. The variety of suitable sensing layers allows the tailoring of the sensor towards the needs of a specific application. Their basic operating principle is that any change in the mass or physical-chemical properties of the film is registered as a frequency shift. These devices operate by detecting surface acoustic waves in the frequency range of KHz up to as high as 600 MHz. We will present some of our recent results that demonstrate the applicability and effectiveness of this technique at high operating frequencies of up to 1 GHz. In many cases a dual SAW set-up is used and the signal of the sensor is mixed with a reference signal from an uncoated SAW to minimize noise and temperature effects. This mixing also lowers the measured frequency.

However, in the last two decades the technologies undergoing dramatic and rapid changes due to the efforts of researchers to overcome the technological hurdles faced by conventional technology. There is also the next generation of sensors systems on which researchers are working.

Spin wave based sensors where a spin wave is used as a physical mechanism for information transmission and processing. These sensor systems may be beneficial in terms of power consumption, resolve the interconnect problem and enhanced logic functionality. This sensor system has potential to detect agents down to the part per billion range.

Graphene based sensor systems are promising for exceptionally high sensitivity (1ppb) due to the unique nature of this strictly two dimensional material that offers a combination of features important for solid state gas sensors. It has essentially only a surface and no volume, which maximizes the effect caused by surface dopants. It is highly conductive, having few crystal defects and exhibiting metallic conductivity even in the limit of zero carrier concentration. It also allows four probe measurements on a single crystal chip with electrical contacts that are purely ohmic and have low resistance. These features combined minimize an intrinsic (excess) noise, which should normally conceal changes caused by individual absorbed dopants.

Furthmore, various branches of new technologies such as MEMS, NEMS and biotechnology have been combined to form new field such as nanobiotechnology. With respect to nanobiotechnology, there is great interest in micromechanical biosensors and nanomechanical biosensors that use the nanomechanical technology of MEMS and NEMS. Micromechanical and nanomechanical biosensors are devices that measure physical quantities by utilizing variations in the physical properties of specifically fabricated microstructure that originate from biological interactions. In micro-cantilever biosensors, the cantilever transduces the recognition event from its receptor-immobilised surface (for example, a DNA probe and an antigen or antibody) into a mechanical response (for example, static displacement and resonance frequency). The mechanical response can then be detected with different methods. Micro-cantilever biosensor has many advantages such as a greatly reduced size, high sensitivity, increased minimum detectable sensitivity and greater reliability. Recent examples of coated cantilevers with TMOs by our lab will be discussed and future prospects will be presented.

## **MICROELECTRONICS BASED BIOSENSORS FOR THE DETECTION OF PROTEINS, NUCLEIC ACIDS AND CELLS**

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Molecular diagnostics is currently focusing on post-symptomatic diagnosis and monitoring of the effect of drugs. In the future, predisposition testing, targeted monitoring, individual therapy selection and follow-up will become key activities in this field. In such a scenario, highly sensitive and specific, yet cheap diagnostic tools will be needed. In addition, they should be rapid, simple, non-invasive and stand-alone if to be used at the point-of-care or point-of-surgery. Miniaturized and integrated lab-on-chip devices that allow genomic profiling and identification of particular biomarkers might fulfill those criteria. However, such systems should dramatically improve on cost, speed, sensitivity, selectivity and ability for multiplexing. Novel biosensor concepts inspired by micro-electronics might allow us to meet these needs.

Novel biosensor concepts are being explored, more specifically for the transduction of affinity-based interactions (*e.g.* antibody–antigen binding and DNA hybridization), into electrical signals. The specificity of affinity-based biosensors depends on the molecular recognition properties of receptors (*e.g.* antibodies) or on the hybridisation of complementary nucleic acids. In this tutorial, various transducer technologies for the detection of affinity-based interactions will be discussed, focusing on the exploitation of microelectronics and nanotechnology. Several examples of label-free detection will be given. These include electrochemical (*e.g.* impedimetric and FET-based) and optical (*e.g.* based on plasmonics) biosensors. The use of labels to enhance the sensor signal will be discussed as well. One particular example where the use of labels has a high added value will be highlighted, *i.e.* magnetic biosensors. There, magnetic particles can be used for sample preparation as well as detection; which allows an entry into the lab-on-chip field.



**SURFACE CHEMISTRY TO BRIDGE INORGANIC BIOSENSOR  
SURFACES AND BIOLOGICAL MATERIALS****Wim LAUREYN***NEXT-NS, IMEC, Leuven, BELGIUM**Contact: laureyn@imec.be*

A key aspect in the realisation of biosensors is the integration of the inorganic (*i.e.* transducer surface) and organic (*i.e.* biological element) components of the biosensor. In this tutorial, various approaches for the realisation of adequate biosensor interfaces will be discussed. One of the most promising methodologies, *i.e.* the use of Self-Assembled Monolayers (SAMs) and crosslinkers to covalently attach the biomolecules onto the sensor surface, will be elaborated into detail.

The increasing miniaturisation of biochips based on microelectronics (and of spot densities in microarrays) and the demand for higher sensor detection sensitivities put severe demands on the process and methodology of coupling biomolecules to surfaces. More specifically, controlled thin film structures have to be created which allow the bio-affinity elements to be arranged and addressed in a reproducible and controlled manner. Addressing these issues, promising methodologies for the construction of novel, well-defined bio-interfaces have been developed, based on the deposition of uniform monolayers of Self-Assembled Monolayers (SAMs) on metal (*e.g.* gold) and oxide (*e.g.* SiO<sub>2</sub> and Ta<sub>2</sub>O<sub>5</sub>) surfaces. In order to retain biological activity (*i.e.* their catalytic or affinity properties) and to allow for the necessary accessibility, the biomolecular functional units have been immobilised onto gold surfaces derivatised using mixed SAMs. In this strategy, the first molecule in the mixed SAM carries a functional group to firmly attach the bioreceptor molecule and the second molecule resists the non-specific adsorption of undesired biological entities. The resulting bio-interfaces can be of significant value for the realisation of highly sensitive, stable and reproducible biochips for a variety of sensing applications.

## **SENSORS FOR MONITORING AIR QUALITY IN EARTH AND SPACE ENVIRONMENTS**

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The monitoring of the quality of air in an enclosed environment has always been an important concern. Toxic gases can be present as a result of leaking in tanks, piping, etc., and their presence has to be monitored to prevent a hazardous condition. Even non-toxic gases such as N<sub>2</sub> leaking in an enclosed area can displace enough oxygen such that a potentially lethal situation can occur.

Advances in nano technology have led to the availability of smaller and more accurate gas sensors, which are being optimized to detect and quantify the presence of a variety of gases. Computer power to process large amounts of data is no longer the prevailing issue; thus multiple and redundant sensors can be used to obtain more accurate and comprehensive measurement. When multiple nano sensors are involved, intelligence can be embedded at the sensor level to limit the flow of nonessential information and to reduce wiring and signal bandwidth requirements otherwise needed to deliver the information. By using distributed intelligence along with relationship rules, complex measurements can be broken down into simpler, smaller ones. Basic knowledge rules incorporated at the sensor allow decision-making capabilities to be decentralized and located at the site being monitored.

The use of small and sensitive nanosensors can allow for the placement of multiple devices over a large area, thus allowing for a more precise and timely determination of a gas leak. Nano sensors are being developed for the detection of various hazardous gases, including but not limited to: H<sub>2</sub>, NH<sub>3</sub>, N<sub>2</sub>O<sub>4</sub>, hydrazine, and others. Testing and development is continuing to improve the response and recovery times, and to increase the sensitivity of the devices. Different coatings and electrodes are currently being evaluated to determine the optimum configuration for the detection and identification of a variety of gases.

The small footprint of the nanosensors allows for several devices, each responsive in a different way to different gases, to be placed on a single substrate. Multiple devices embedded on a single substrate result in increased reliability and in a decrease in the frequency of periodic calibrations. The use of different coatings for individual elements of a multi-channel sensor allows for the identification of different gases.

Monitoring of the environment is not limited to the detection of gas leaks, and it will become increasingly important as longer duration space missions are planned and executed. A multitude of sensors need to be developed and qualified for space flight under realistic operating conditions, and as such, development activities in the nano sensor field will continue for years to come.

This lecture will describe the newly available nano sensors as well as a methodology to process information in real-time to allow for the monitoring of air quality.

**SENSORY CONVERSION DEVICES****Pedro J. MEDELIUS**

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The human body has five basic sensory functions: touch, vision, hearing, taste, and smell. A broad definition of a sense would be "a system that consists of a sensory cell type (or group of cell types) that respond to a specific kind of physical energy.

The effectiveness of one or more of these human sensory functions can be impaired as a result of trauma, congenital defects, or the normal aging process. Thus converting one type of function into another, or translating a function to a different part of the body, could result in a better quality of life for a person with diminished sensorial capabilities. For instance, it is possible to transfer a sensorial function such as touch from an impaired part of the body to a fully functional part by means of sensors and nano/micro actuators. The availability of newly developed nanotechnology-based sensors, fast signal processing capabilities, and low power electronics, is enabling the conversion of sensory functions.

The following table illustrates a few of the possible sensorial conversions that can be implemented with newly developed sensors and display mechanisms:

<b>Diminished Function</b>	<b>Conversion Device</b>	<b>New Sensorial Function</b>
Touch	Electronic skin sensitive to temperature, pressure, shock,	Visual indication Audible indication: variable tones Temperature & pressure actuators on able parts of the body
Vision	CCD Imager	Audible description of objects in the field of view: real-time pattern recognition  Actuators for the representation of images, color, depth, etc. as a combination of pressure and vibration on the skin
Hearing	Stereo microphones	Real-time visual display: voice recognition and direction-of-arrival information
Smell	Electronic nose	Visual indication Audible indication

This lecture will concentrate on the description of available nano and micro sensors, the means to process the information from the sensors in real-time, and the optimization of the conversion and display mechanisms.

## **OPTICAL SENSORS FOR CARBON DIOXIDE AND THEIR APPLICATIONS**

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There are few analytes in the world as significant as carbon dioxide, since it is a basic chemical feedstock of life which, when coupled with green plant photosynthesis, generates the fuel and food necessary for the continued existence of most known forms of life. Most cell metabolism is associated not only with the release of energy for life but also carbon dioxide. Thus, not only is carbon dioxide usually an essential ingredient to make the prerequisite chemicals for life, via photosynthesis, it is also often used as an indicator of the existence of life and a measure of health, via respiration. Not surprisingly, therefore, in medicine, the key basic analytes that are routinely monitored in the blood of hospital patients are: dissolved oxygen, pH and carbon dioxide.

In clinical chemistry, a whole area devoted to the monitoring of the levels of carbon dioxide in breath has emerged in recent years, i.e. capnography, in which not only the level of carbon dioxide is important, but also its temporal variation, since both provide valuable medical diagnostic information. Not surprisingly, the measurement of carbon dioxide is also an important feature of environmental monitoring, providing, as it does, a rough gauge of the health of the hydrosphere or atmosphere. The use, presence and measurement of carbon dioxide is also important in many industries, including brewing. In the food industry, a revolution in food packaging has come about through the use of carbon dioxide in modified atmosphere packaging (MAP). In many industries, the use, or presence, of carbon dioxide is commonplace and its measurement and continuous monitoring often essential.

In this talk the basic concepts behind the major, and some minor, different colourimetric and luminescent optical sensors for the detection and quantitative analysis of carbon dioxide will be reviewed. Most, but not all, are based on the pH effect of carbon dioxide on the medium surrounding an indicating dye. Examples and the characteristics of the different indicators are given, along with their key advantages and disadvantages. The major applications of these sensors will be discussed and examples of their commercial application given.

**OXYGEN INDICATORS IN FOOD PACKAGING****Andrew MILLS**

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The detection of oxygen is important and optical sensors for this purpose are of increasing interest, especially in modified food packaging (MAP), in which the food package is flushed with a gas, such as carbon dioxide or nitrogen. An oxygen optical sensor for MAP should be inexpensive, rapid-responding and irreversible. The reasons behind the need to develop such a sensor for MAP are discussed.

The different types of indicator that have been developed to date fall mainly into the following categories: reversible luminescent indicators, reversible colourimetric indicators and visible and/or UV light activated indicators. The basic technologies underpinning each of these three different indicator types are described, their various typical components listed and their roles described. Examples and the characteristics of the different indicators are given, along with their key advantages and disadvantages. The potentials of these different indicators for application in MAP are evaluated briefly. Particular attention is given to a new irreversible, reusable, colourimetric, UV-activated oxygen indicator that shows great potential.

Brief details of the underlying techniques used to support the above indicators will be reported including: time-resolved luminescence lifetime measurements, fluorimetry and UV/Vis spectrophotometry. The lecture aims to provide a comprehensive introduction into the rapidly developing realm of oxygen indicators in food packaging.

**ADVANCED NANOMATERIALS FOR SENSING****Sudipta SEAL**

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Materials are an integrated component in sensor development. Gas sensors based on various mechanistic principles, such as Schottky diode, optical, metal nano-wire, solid and polymer electrolyte, thermoelectric, surface acoustic wave, micro-cantilever, diamond-quartz crystal microbalance, thermal conductivity, and resistance based, have been investigated in the literature. In this presentation, we review the current understanding of the working principle of the semiconductor oxide sensors based on resistance-change mechanism. Various material fabrication techniques, such as, such as spray pyrolysis, chemical vapor deposition, ion assisted deposition, sputtering, evaporation, arc plasma, metal organic deposition, and sol–gel are used for sensor material development. Solgel processing, a cost effective synthesis technique is discussed in the development of advanced nanomaterials (primarily semiconducting oxides) for sensing.

**NANOSENSORS****Sudipta SEAL**

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In the recent years, a significant interest has created nanostructures for the detection of gas, volatile organic compounds (VOCs), and bio-molecules in the wake of counter-terrorism threats and environmental pollutants. The enhancement in the selectivity and the overall efficiency of the sensor is achieved by tailoring the size, the structure, and the shape of the nanoparticle integrated in nanosensors. In the last decade, numerous studies revealed various sensing properties of these nanomaterials. However, there have been improvements in overall sensor performance; cross selectivity and robustness remain a problem. This warrants a proper understanding of the basic principles behind the sensing properties in nanosensors. Various aspects in the development of nanosensors are presented herein.

## LIGHT-INDUCED GENERATION OF SINGLET OXYGEN IN POROUS SILICON

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It is well known that molecular oxygen is normally in the ground state  $^3\Sigma$ , which is triplet one. The excited states of oxygen molecules are singlet ones and they are characterized by the energy excess of about 0.98 eV ( $^1\Delta$ ) and 1.63 eV ( $^1\Sigma$ ) in respect to the ground state [1]. Nowadays, huge oxidizing properties of the singlet oxygen (SO) are widely used in the photodynamic therapy (PDT) of cancer. Despite the selection rules do not allow the efficient optical excitation of molecular oxygen, SO can be generated by using a photosensitizer [2]. It was demonstrated that Si nanocrystals assemblies, i.e. porous silicon (PSi), act as an efficient photosensitizer of SO [3-5]. The photoluminescence (PL) properties of PSi are well explained by the radiative recombination of excitons confined in Si nanocrystals [6]. The excitons can non-radiatively transfer their energy to oxygen molecules adsorbed on Si nanocrystal surfaces [3]. The energy transfer is usually observed as a quenching of the exciton PL, which is maximal at the wavelength of 760 nm, i.e. at 1.63 eV ( $^3\Sigma$ - $^1\Sigma$  transition in oxygen molecules) [3]. The PL spectroscopic experiments clarify that the energy transfer is mediated by a resonant direct electron exchange between photoexcited Si nanocrystals and oxygen molecules [4]. This process was shown to be also possible for PSi dispersed in oxygen-saturated benzene [5]. For applications in PDT, the SO formation in aqueous solutions is obviously required. Recently, the photosensitization of SO by PSi dispersed in water has been demonstrated and the corresponding biomedical applications in PDT have been proposed [6]. PSi samples were prepared using the conventional method of the electrochemical etching of c-Si wafers in hydrofluoric acid solutions. The obtained free-standing PSi films were dried in air and then milled to get a powder with the maximal size of PSi pieces of the order of 1  $\mu\text{m}$ . Then the PSi powder was dispersed in pure water bubbled with oxygen to get a homogeneous aqueous suspension. The PL spectra of PSi were detected using a CCD chamber, the PL transients were registered using a photomultiplier and recorded by a digital oscilloscope. For PDT experiments we employed cancer cells of 3T3 NIH (mouse fibroblast) grown by standard procedure of their sub cultivation *in vitro* in the Dulbecco-modified Eagle's medium using 96-well plates or dishes. The cells were cultivated during 1.5 days, then the medium was changed on the fresh nutrient solution and after this the PSi suspension with different concentrations of Si nanocrystals were added to the cells. One part of the wells was illuminated by mercury lamp, another part was kept in darkness and some cells were used for control (without PSi and illumination). The inhibitory or lethal effect

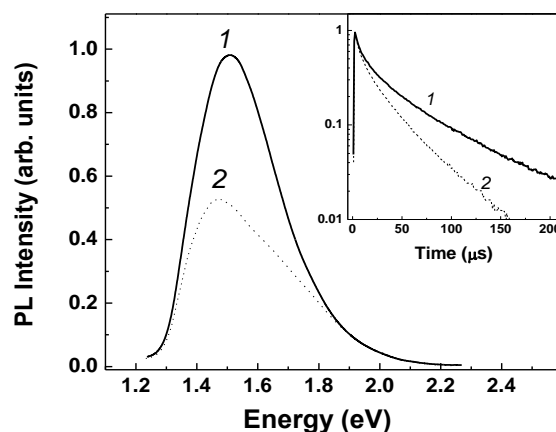


Fig.1. PL spectra of PSi dispersed in oxygen-free water (1) and in water, saturated by oxygen at 1 bar (2). Inset: transients of the PL at 1.63 eV of PSi dispersed in oxygen-free water (1) and in water with oxygen at 1 bar (2).



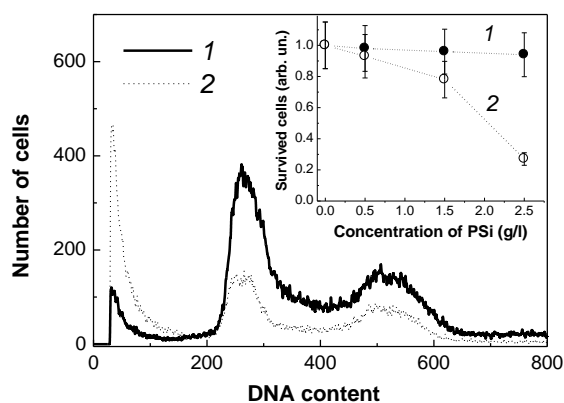


Figure 2. Typical histograms of DNA content for the cancer cells of mouse fibroblasts kept in the nutrient solution with dispersed PSi (1.5 g/l) in darkness (1) and after illumination (2). Inset shows the relative contribution of the survived cell number vs the concentration of PSi in darkness (1) and after illumination (2).

from 2 to 4 nm [7]. The PL intensity in oxygen-saturated water decreases in comparison with that in oxygen-free water (or in vacuum). The spectral maximum of the PL quenching lies at 1.63 eV. This fact proves that the PL quenching is caused by the energy transfer from excitons in Si nanocrystals to  $O_2$  molecules, i.e. by the SO photosensitization. Additional evidence of this process can be obtained from the PL transients. The inset of Fig.1 depicts the exciton PL transients at 1.63 eV for PSi dispersed in oxygen-free water and in oxygen ambient. The PL lifetime decreases for the PSi in oxygen-saturated water. From the analysis of the lifetimes and efficiency of the exciton PL we conclude that at least 40% of the excitons in Si nanocrystals transfer their energy to the dissolved in water  $O_2$  molecules.

Dependence of the cancer cell number (relative to the control) as a function of the PSi concentration in the solution is given in the inset of Fig.2. One can see that the number of cells decreases strongly after illumination. This fact is concerned with the SO generation by dispersed PSi. The cell cycle analysis was performed to confirm the death of the cells in illuminated dishes. It should be noted that cells with the smallest DNA content (typically less than 100) can be considered as apoptotic cells (dead due to the apoptosis mechanism) [8]. One can see from Fig.1 that the illumination of the cells with added PSi suspension leads to an increase of the distribution peak at low DNA content, which means an enhancement of the apoptosis rate because of the SO generation. These results demonstrate good prospects for the biomedical applications of PSi.

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of the illumination was obtained by measuring the cell quantity in non-illuminated and illuminated wells per cells quantity in the control wells in which the PSi-suspension was not added. For this purpose a method of cells staining by crystal violet was used. Additionally, the cell cycle analysis was performed by using a flow cytoflourymeter (see for details Ref.[6]).

Typical PL spectra of the PSi powder in water are shown in Fig.1. The PL spectrum is characterized by a broad band centered at 1.5 eV and it can be interpreted as the radiative recombination of excitons confined in Si nanocrystals with sizes distributed

## SINGLET OXYGEN GENERATION AND DETECTION FOR BIOMEDICAL APPLICATIONS

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Molecular oxygen is one of the most important substances on the earth. Almost all living organisms utilize oxygen for respiration and energy generation. In 1840 Michael Faraday discovered that molecular oxygen is attracted to a magnet. Almost a century later, in 1925, Robert Mulliken explained why oxygen is magnetic using the recently developed quantum theory. In 1931 Hans Kautsky in Heidelberg University discovered an active form of molecular oxygen – singlet oxygen (SO). According to the quantum theory, the spin configuration of the lowest energy state with unpaired electrons in two different molecular orbitals accounts for the paramagnetism of molecular oxygen in the ground state (triplet oxygen), which is labeled as  $^3\Sigma$  (here and afterwards subscript denotes degeneracy of the state). The excited states of oxygen molecules are singlet ones and they are characterized by the energy excess of about 0.98 eV ( $^1\Delta$ ) and 1.63 eV ( $^1\Sigma$ ) in respect to the ground state [1]. The transition from the triplet state to the SO ones requires spin-flip process and the direct states conversion via absorption of photons is spin-forbidden in the first approximation. Since the photon absorption and emission are mirror-like processes the pure radiative decay lifetimes of  $^1\Delta$  and  $^1\Sigma$  states are extremely long: 45 min and 7 sec respectively.

SO mediates important processes in chemistry and biology. It reacts with many organic compounds including aromatics, steroids, fatty acids, vitamins, amino acids, proteins, nucleic acids and synthetic polymers. It is involved as well in modification of biological structures. Some examples are lipid peroxidation, photohemolysis and most important photodynamic therapy combining drug and light treatment of malignant tumors. A several methods of SO production have been developed so far. Thermal and gaseous discharge methods are not compatible with application requirements since the excitation process requires high temperature or accompanied by generation of oxygen atoms/ozone, respectively. The most useful and widely used generation procedure includes a second substance called “photosensitiser” which is commonly a strongly absorbing organic dye molecules or fullerenes. It was found that silicon nanocrystals act as efficient photosensitizers of SO due to the resonant electronic energy transfer from excitons. A quenching of the exciton photoluminescence can be used to measure the SO generation efficiency. Furthermore, the electron paramagnetic resonance (EPR) techniques can be used to study the photosensitization of SO on surfaces of Si nanocrystals. EPR spectroscopy can detect both Si dangling bonds on nanocrystal surfaces and their interaction with adsorbed oxygen molecules in the ground (triplet) state. A strong decrease of the spin relaxation time of Si dangling bonds is observed under illumination of the nanocrystals in oxygen atmosphere and it can be employed to monitor quantitatively the SO photosensitization. The dependences of the SO concentration on mean size of Si nanocrystal, partial pressure of oxygen, and illumination intensity are experimentally obtained and discussed in view of possible biomedical applications, i.e. photodynamic therapy of cancer.



## **NANOMATERIALS IN ENVIRONMENTAL POLLUTION DETECTION, MONITORING, AND REMEDIATION**

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Pollution poses a major challenge all around the world, adversely affecting the lives of millions of people by contributing to debilitating and deadly health disorders. Pollution, in general, is contamination by a chemical or other agent that renders part of the environment unfit for intended or desired use. Natural processes release toxic chemicals into the environment as a result of ongoing industrialization and urbanization. Recent studies on the effects of chronic exposure to air pollution have singled out particulate matter as the most responsible pollutant for the life-shortening effect, although other pollutants may also play a vital role. Furthermore, contaminants in soil enter into the watershed to further exacerbate wide spread pollution. Consequently, rapid detection of contaminants in the environment, such as air, soil, and water streams by emerging technologies is of paramount significance. Environmental pollution in some developing countries have reached alarming levels and hence real-time pollution monitoring sensors, sensor networks, and real-time monitoring stations need to be employed to gain a thorough understanding. A tool providing interactive qualitative and quantitative information about the pollution is essential for policy makers to implement policies to protect massive populations, especially in developing countries. The objective of this investigation is to utilize nanoscale materials, devices, and systems as in-situ sensors and detectors of chemical and biological agents coupled with remote satellite image processed data for environmental air pollution monitoring. The efficacy of nanoparticles based environmental sensing using commercially available nanoscale metal-oxide based gas detectors is augmented by image processed satellite data to monitor local and regional air pollution dispersion. Using nanotechnology based portable, wireless and web-based gas sensors; pollution is monitored at several ground stations. Employing remote sensing technologies with high spatial and spectral resolution and image processing, we model urban pollution to quantify and authenticate the ground based data. One of the objectives of this investigation is to develop a unique capability to acquire, display and assimilate these valuable sources of information to accurately assess urban pollution by real-time monitoring using commercial sensors fabricated using nanofabrication technologies and satellite imagery. In addition to air pollution, we explore the efficacy of nanostructured materials in the detection and remediation of water pollution. We present our results of sorption on advanced nanomaterials based sorbents that have been found effective in the removal of cadmium, arsenic, and degreasers such as trichloroethylene from water streams.

**DYNAMICS AND TRANSPORT OF NANOMATERIALS  
IN THE ENVIRONMENT AND HUMAN HEALTH IMPLICATIONS  
AN ONTOLOGICAL MODALITY**

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Nanoscale materials, devices, and systems are at the confluence of the smallest engineered materials/devices and the largest molecules of the biological systems. Advances made over the last few years provide new opportunities for scientific and technological developments in nanostructures and nanosystems with new system architectures with improved functionality. Despite of major developments in this field, there is a significant gap in our knowledge of the environmental, health, and ecological impacts associated with nanotechnology. Since innovations in the field of nanotechnology occur faster than the policymakers can develop safe handling practices; a comprehensive and fundamental investigation is necessary based on dynamic transport of nanomaterials and its impact on human health and ecology. A matrix of parameters which govern fate and transport of nanomaterials such as exposure routes, chemical composition, surface structure, solubility, size and shape effects, toxicity, absorption, distribution, metabolism, agglomeration, and excretion rate and mechanisms is proposed in this investigation. The complex nature of naturally occurring and engineered nanomaterials and transport either in the environment or via different exposure routes with human body necessitate an ontological modality. The essential aspect of employing ontology is in defining not only the item, but also its relationship to other items in the glossary, citing example from ontological approaches in computer science advances, namely the ability to retrieve information from various sources and databases. A theoretical foundation and basis for ontologies used for fate and transport of nanomaterials in the environment such as air, water, and soil and human body will be presented. Biodegradation and bioaccumulation of nanomaterials have not been addressed in the literature despite the significant impacts they pose on human health. A comprehensive investigation will prove beneficial to risk assessment and ensuring safe practice in nanotechnology.

## MIS-STRUCTURE SENSORS WITH SOLID ELECTROLYTE GATE LAYER FOR THE DETECTION OF EXPLOSIVE AND HAZARDOUS GASES

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In this presentation we consider the application of MIS structure gas sensors with solid electrolyte layer (MEIS structure) for the detection of concentrations of a number of hazardous gases, which can be dangerous themselves (fluorine, hydrogen fluoride, probably - H<sub>2</sub>S, NO<sub>2</sub>, etc.) as toxic agents or are important from ecological point of view (for example, chlorofluorocarbons, which destroy ozone layer and are responsible for green-house effect). The third class of gases like hydrogen is indicator of early stage of fire (smoldering) and is used for prevention of hazardous situation and, therefore, loss prevention.

MEIS sensors have a structure Si(SiC)/SiO<sub>2</sub>/solid electrolyte/Pt(Pd). Solid electrolytes, which can be used in this case are LaF<sub>3</sub> (F<sup>-</sup> conducting electrolyte) or various proton conducting materials. Recently, the possibility of the application of some ion-exchange materials is also under study. This sensing structure enables the separation of two processes responsible for gas sensing: electrochemical reaction on three-phase interface gas/Pt/solid electrolyte and ionosorption on the solid electrolyte/SiO<sub>2</sub> interface. These two interfaces, which are the main working elements of the sensor, play different roles. The first acts as a reversible electrochemical electrode with respect to target electrochemical reaction of the formation of fluorine ions in the case of fluorine conducting electrolyte or protons in the case of proton conducting material. At the same time, the second interface is a blocking electrode, therefore the ionosorption process on this electrode is fast compared to processes on Pd/SiO<sub>2</sub> or Pd/Si<sub>3</sub>N<sub>4</sub> interfaces typical of hydrogen MIS structure sensors with Pd gate. As a result, the CV-characteristics of the MEIS structure shifts along voltage axis, in general, in accordance with Nernst law and the value of the voltage shift is proportional to the logarithm of gas concentration. The separation of processes accelerates significantly overall process compared to usual hydrogen MIS-structure sensors with Pd gate and extends concentration range.

As a result, the sensors based on MEIS structures with solid electrolyte layer have several important advantages compared to other types of sensors: they operate at room temperature, are produced using Si-compatible technology, and, therefore, can be applied in mass-application instruments including wireless networks.

Detection of fluorine and HF with MEIS structures using LaF<sub>3</sub> solid electrolyte. The sensing structure was fabricated by the thermal evaporation of lanthanum trifluoride on the surface of Si covered with SiO<sub>2</sub>/Si<sub>3</sub>N<sub>4</sub> (80/20 nm). The thickness of solid electrolyte layer was of about 200 nm. Platinum gate electrode was deposited by Pt sputtering in argon atmosphere in diode sputtering system. It was shown that the sensitivity to fluorine concentration in synthetic air is equal to 30 ± 2 mV per decade of fluorine concentration at room temperature. This sensitivity

corresponds to a simple two-electron process taking place on the three-phase interface gas-platinum-solid electrolyte. An increase in the working temperature of the sensor changes completely the electrochemical mechanism of the sensor response; it does not correspond anymore to simple electrochemical process. This response type was observed in a concentration range below approximately 100 ppm of fluorine. At higher concentrations, or at the application of gold gate metallization, or after preliminary treatment at 150°C in air the sensor sensitivity is not constants and can reach 120 mV per decade. This effect is due to the formation of fluorides on the surface of metal gate. HF sensitivity of the sensor at room temperature is equal to about  $45 \pm 2$  mV per concentration decade. This value of sensitivity is due to the formation of mixed potential. The main problem met at the detection of hydrogen fluoride with MEIS sensors is the formation of lanthanum oxifluoride on the surface of lanthanum trifluoride. This effect leads to the “sleeping” of the sensor after long storage in humid air. Therefore, the minimum concentration of hydrogen fluoride that should be applied to the sensor to reactivate it is equal to about 5 ppm. After this, the sensor can be used for the detection of low concentration below MAC level (0.5 ppm).

Detection of fluorocarbons. In this case, we used high-temperature MIES structure based on SiC (Pt/LaF<sub>3</sub>/SiO<sub>2</sub>/SiC/Ni). Optimum working temperature of the sensor is of 300 – 500°C depending on particular fluorocarbon to be detected. It was shown that the detection mechanism of the sensor to fluorocarbons is due to direct interaction of these gases with platinum-solid electrolyte interface. The sensitivity of the MIS structure gas sensor to different fluorocarbons is in a range between 10 and 20 mV per concentration decade. The limit of detection of fluorocarbons is of about 100 ppm. The main problem met at the application of SiC based sensors is common for all high-temperature silicon carbide sensors. This problem is stability of Ni- or W-made ohmic contact to SiC at high temperature.

Detection of hydrogen. The detection of hydrogen is possible at room temperature with the application of both fluorine and proton conducting solid electrolytes. Lanthanum trifluoride based sensors can be applied after the procedure of sensor reactivation. This reactivation consists in the heating of the sensor in air up to ~ 170°C for short time (me tested mainly the heating during ~ 1 min). This heating leads to the formation of a layer of lanthanum oxifluoride on the interface of platinum with lanthanum fluoride. This layer operates as a reversible electrode with respect to both oxygen and fluorine. Therefore, the sensor becomes sensitive to hydrogen concentration. The reactivation procedure should be repeated every several hours, because the layer is not stable. This regime enables the measurement of hydrogen concentrations in a wide concentration range from approximately 100 ppb to 100%. The possibility to measure high concentrations of hydrogen is a very important difference of this type gas sensors from the MIS structure sensors with palladium gate and without solid electrolyte layer. This last sensor is saturated at high hydrogen concentrations.

The application of proton conducting solid electrolyte layer is very promising for the application of hydrogen sensors in gas fire detecting systems. We tested the response of the sensor with NAFION layer, the layer of zirconium hydrophosphate, and with layer of new prospective solid electrolyte – ester of polyvinyl alcohol with tungsten heteropolyacids. The sensitivity of all sensors except ester electrolyte layer is close to 120 mV per concentration decade. This high sensitivity is due to the formation of mixed potential on the gas – platinum – solid electrolyte interface. The drawback, which should be improved in future, is the stability of the polymeric solid electrolyte layer. This problem could be solved by the application of cross-linked polymers based on esters of polyvinyl alcohols with tungsten heteropolyacids.

## SENSORS BASED ON TECHNOLOGY “NANO-ON-MICRO” FOR WIRELESS INSTRUMENTS PREVENTING ECOLOGICAL AND INDUSTRIAL CATASTROPHES

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Last decades were characterized by very strong development of new tools for information processing. However, the attention paid to the fabrication of instrument used for data and information acquisition was not sufficient. In particular, this concerns the development of devices designed for the accumulation of data about chemical composition of gases and liquids (chemical sensors) compatible with microprocessor-based data processing instruments. This compatibility requires low power consuming and low cost (“Smart Dust”) of devices applicable in distributed wireless nets used for prevention of different dangerous situations (explosions, fires, gas leakage, etc.).

From technical point of view, the problem of gas analyzers compatible with wireless nets can be solved by the application of sensors based on the technology “nano-on-micro”. The idea of this technology consists in the application of nano-structured and nano-composite sensing materials of metal oxide semiconductor of thermocatalytic layer deposited on microhotplate fabricated using silicon or alumina microelectronic technology. As a result, the sensor combines the advantages of both technologies – high stability and sufficient selectivity of nano-composite materials and, on the other hand, microprocessor compatibility, low-cost, mass-production possibilities, and low power consumption of microelectronic substrate.

Two methods of the fabrication of microhotplates are most promising: silicon based technology of silicon oxide/silicon nitride membranes and the technology of thin alumina films (TAF). In both approaches, the membrane stretched on the rigid frame made of bulk material (silicon wafer or alumina ceramics, respectively) is used as a support of functional elements of sensor (heater, thermometer, and sensing layer). On the other hand, thin membrane having low heat conductivity in superficial direction is used for thermal isolation of hot sensing area of the sensor.

The optimization of the layout of the microhotplates fabricated using silicon technology showed that the optimum of sensor power consumption is observed, when the heat losses due to thermal conductivity of the membrane and through ambient air are approximately equal. At temperature below 500<sup>0</sup>C typical of semiconductor gas sensor and for small sensing element (~200 microns) radiation and convection heat losses are negligible. These conditions are met for 200 micron microheater, if the membrane to heater size ratio is of about 8 – 10, that is the membrane size should be of about 1.5 – 2 mm. The main problems restricting mass-application of such microhotplates are the accumulation of internal stresses in the multilayer membrane consisting of a stack of silicon oxide and silicon nitride and poor adhesion of pure



platinum to silicon oxide or silicon nitride upper layer of the membrane. The first problem leads to the destruction of the membrane after temperature cycling and even after long-term operation at constant temperature. The second problem requires the application of adhesive layers deposited between silicon oxide and platinum. This layer (Ti, Ta, or Cr) can be easily oxidized at temperature exceeding  $\sim 400^{\circ}\text{C}$ . Therefore, platinum heater peels off from the membrane after annealing and during the work. This problem restricts the application of sensing layer deposition on whole wafer, because sensing layer needs a stabilization annealing at high temperature.

A possible solution of these problems is the application of thin alumina film (TAF) membrane MEMS platforms for gas sensor fabrication (CeraMEMS). Alumina TAF membrane, (20 – 30 microns thick) is fabricated by electrolyte spark oxidation of aluminum. At several hundreds volts, the dielectric layer on the electrolyte/metal boundary is broken, and results in micro-sparks. In this way, the oxide layer grows. The spark's high temperature forms a uniform, polycrystalline  $\alpha$ -modification oxide (crystallite size of about 20 nm) on the surface of metal. The membrane is separated from Al by wet etching and fixed on ceramic wafer with holes drilled preliminarily using laser beam. The holes were 3 mm in diameter. The meander shaped heaters were produced by platinum sputtering through shadow mask. Heater size of 300 microns and membrane diameter of 3 mm correspond to minimum power consumption of the sensor. Platinum coating was optimized taking into account long term stability of platinum heater working under harsh conditions and at temperature up to  $600^{\circ}\text{C}$ . Resulting resistance of the heater was in a range of 10 – 30 Ohm. Each substrate (48 x 60 or 100 x 100 mm) contained from 80 to 400 individual chips.

Advantages of TAF platforms are: (1) TAF membranes enable the work at temperature up to  $600^{\circ}\text{C}$  and, prospectively, up to  $800^{\circ}\text{C}$ ; (2) the TAF microhotplate is more robust than silicon chip with thin membrane; (3) in contrast to silicon oxide, platinum adhesion to alumina is perfect without the application of any adhesive layers; (4) the middle scale production of these chips ( $10^4 - 10^7$  chips per year) is cheaper compared to silicon technology.

TAF CeraMEMS platform was successfully used for the fabrication of semiconductor and thermocatalytic gas sensors operating in pulsing heating mode, as a source of IR radiation for optical gas sensors and as bolometers. Maximum frequency  $\sim 10$  Hz (thermal response time  $t_{0.63}$  is of 80 ms). The sensor can withstand  $\sim 7 \cdot 10^6$  on-off cycles sufficient for 3-year operation of  $\text{CH}_4$  sensor, average power consumption of the sensor can be below 1 mW. Heater resistance drift  $< 3$  % per year at  $550^{\circ}\text{C}$ .

The main applications of “nano-on micro” sensors are the determination of the concentrations of gases produced in the very initial stages of fire (smoldering) and prevention of ecological or industrial catastrophes. Among the gases, which could be detected are usual gases like methane, hydrogen, carbon monoxide, etc. These sensors are used also for the determination of concentration of gases resulting from terrorist attacks.

The sensors designed using both approaches are applied in autonomous and wireless systems. This is possible because of extremely low average power consumption of the sensors – less than 1 mW at duty cycle of about 1 %. A novel data processing method based on temperature modulation enables the detection of a set of gases (for example,  $\text{H}_2$ ,  $\text{CO}$ ,  $\text{CH}_4$ , humidity) with the same sensor. This makes possible further decrease in power consumption compared to usual electronic nose. The combination of modern technologies of fabrication and of data processing enables the production of low-cost mass application instruments.

# ***BOOK OF ABSTRACTS***

## ***2. Seminars***



## PHOTONIC SENSORS FOR HEALTH AND ENVIRONMENTAL MONITORING

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This paper focuses on new trends for photonic sensors with applications in health and environmental monitoring. Photonic sensors are under extensive development world-wide combining multidisciplinary research in e.g. biology, chemistry, physics, electronics and optics. Small, inexpensive, sensitive, selective, fast, robust, and remotely controllable sensors that are immune to electromagnetic interference are desired for applications such as environmental monitoring, healthcare, and for defense, chemical, and bio-agent detection. Different photonic sensor designs have demonstrated the potential to achieve these capabilities. Progress in materials, light sources, photo-detectors, and innovative solutions have driven the technology further.

New sources are important for development of photonic sensors. An example is absorption spectroscopy based sensors that utilize the unique absorption signatures of chemical compounds. Most compounds have their fundamental vibrational modes in the mid-IR. Earlier tunable fiber lasers suffered from narrow tuning ranges and had limited wavelength availability, especially in the mid-IR. This limited the compounds that could be detected. Today laser diodes and quantum cascade lasers have broad tuneability and cover wavelengths from ~630nm to 30μm. This opens for new sensor applications in medical diagnostics (e.g. breath analysis) and for detection of biohazards, drugs, explosives, gases, and chemicals.

Photonic integrated polymer components can potentially be fabricated at low costs using deep UV-induced refractive index modification. Monolithic integration of polymer waveguide structures in optical or fluidic microsystems is possible by combining with replication techniques. Environmental, chemical and biological sensors are envisaged by hybrid integration with organic or inorganic photodiodes, which will drive their costs down.

Silicon photonics using CMOS technology is another candidate to create low-cost, integrated optical components and sensors. An obstacle for mass production is the lack of an electrically driven laser that can easily be integrated in silicon. Today prefabricated lasers are integrated with silicon one at a time. Recent advances in hybrid III-V silicon lasers are promising.

A potentially low cost silicon sensor is being developed for bio-agent detection and air pollution monitoring. This MOEMS sensor exploits Si-based suspended micro-bridge structures. The source has a smart design using photonic bandgap structures with narrow band IR emission to provide high chemical selectivity and sensitivity.

By utilizing the dispersive properties of photonic crystals the size of optical sensors can be reduced. One recent example is based on a Mach-Zehnder interferometer (MZI) typically used for detection of gases.

Nanostructured and nanocomposite materials including polymers, sol-gel materials, crystalline and amorphous metal oxides, and metals are being developed for biomedical and gas sensors. One innovative example is a detector based on a porous silicon chip that changes color when exposed to nerve gas. The chip is made from flexible, biocompatible polymers and has the optical properties of a photonic crystal. Another example is the use of gold nanoparticles laced with DNA to achieve a sensitive and selective colorimetric biosensor for detection of contaminant metal ions. Yet an example is a near-infrared nanoscale sensor that detects glucose based on protein encapsulated single-walled carbon nanotubes (SWCNTs).

Photonic crystal fibers (PCF) or microstructured fibers have attractive properties for sensing such as tolerance to temperature change and high birefringence. Other interesting types of fiber being pursued for sensing are random hole optical fibers (RHOF) and hybrid ordered random hole optical fibers (HORHOF).

Innovative fiber optic sensors are being developed combining e.g. molecular biology and chemistry. Examples are diagnosis of prostate cancer using a polymer gel sensing network that reacts with the target molecule, and detection of organic solvent vapors based on surface plasmon resonance spectroscopy.

Biosensors detect biomolecular interactions. Novel designs open for a multitude of applications. Biosensors based on total internal reflection fluorescence (TIRF) are used to detect analytes such as toxins, bacteria and viruses. Biochips are an array of biosensors that can have different bioreceptors and thereby detect multiple analytes. Bio-chips based on spectroscopy of surface plasmons on an array of miniature diffraction gratings have been demonstrated.

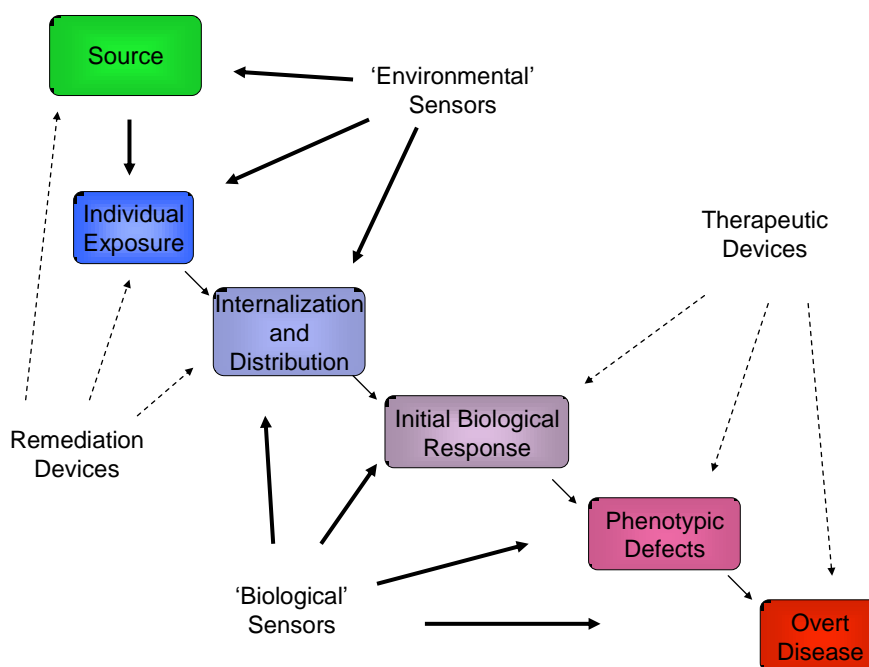
Our paper will demonstrate how sensor designs based on integrated optics, micro-opto-electro-mechanical systems, photonic crystal and photonic bandgap structures, nanomaterials, lab-on-chip, and microfluidics can offer attractive alternatives overcoming both technical limitations and cost issues associated with current sensors on the market.

## SENSOR TECHNOLOGIES FOR LINKING INFORMATION ON EXPOSURE, EARLY BIOLOGICAL RESPONSE AND DISEASE PROGRESSION

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The National Institute for Environmental Health Sciences focuses on investigating the mechanisms by which environmental factors contribute to the initiation, progression and prognosis of human diseases. This broad vision requires an integration of information across multiple levels from individual exposure to internalization and distribution, early biological responses and ultimately the progression of diseases (Figure). The recent advances in micro- and nanoscale engineering offer the potential for developing tools to advance our understanding at each level of this cascade through the development of improved sensor and intervention devices.



**Figure** A schematic representation of a cascade events leading from environmental exposure to disease. Solid arrows represent points along the cascade where sensor technologies can provide information to scientists and clinicians, dashed arrows represent points along the cascade where nanotechnology derived devices offer potential for intervening to halt the development of disease. Adapted from a National Research Council report.

In February 2006, the National Institutes of Health announced a new trans-NIH initiative, the Genes and Environment Initiative, to understand the complex interplay between genetic and environmental factors in human disease. A four year developmental period was outlined that focuses on the development of the resources and methodologies needed for a concerted, large scale population based effort. A key component of this activity is an Exposure Biology Program consisting of five solicitations on developing sensor technologies for assessing personal exposure to toxicants, assessing diet and physical activity, assessing psychosocial stress and the use of drugs of abuse and identifying markers of the biological response to all of the above.

All of these potential applications are enabled by the efficiency that results from decreasing the scale of monitoring. There are several well known advantages to decreased scale, most notably in the reduction of sensor size and weight which enable analyses of multiple analytes simultaneously and decrease the consumption of reagents, decrease power demands and allow the integration of additional capabilities such as wireless telemetry and global positioning. These advantages are further magnified if the scale decreases to the nanoscale, which is defined by the National Nanotechnology Initiative as having a single spatial dimension on the order of 1 to 100 nm. At these scales unique chemical and physical properties emerge, primarily as a result of quantum confinement, which allow for improved sensing based on novel optical, electrical and magnetic modalities. These same properties also enhance the reactivity of nanomaterials, making them ideal for catalytic and chelating approaches to environmental remediation and also as multifunctional therapeutic agents.

This presentation will focus on the applications of micro and nanoscale sensors in the environmental health sciences with an ultimate goal of utilizing these new technologies to unravel the mechanisms by which environmental factors influence human disease. This vision includes an ultimate intention to integrate these sensor technologies with intervention modalities for the creation of 'brilliant' sensors that can detect an exposure, identify the biological response to that exposure, assess whether it is compensatory or adverse, and intervene to halt the progression of disease at the earliest stages. Examples of specific NIEHS supported research will be presented as well as opportunities for further work.

## NEAR FIELD COMMUNICATIONS FOR SENSING AND SECURITY

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Far field wireless electronic systems are increasingly deployed for remote sensing and condition monitoring applications. While wireless monitoring systems undoubtedly offer considerable convenience, they generally offer insignificant improvement in terms of metrology. This paper presents an overview of inductively coupled telemetry, a technology which enables wireless metrology from environmentally extreme and difficult-to-access places, without the need for conventional power sources; such as batteries and generators.

The technology relies upon the transmission of both power and data between two weakly coupled coils, (a transmitter and a receiver), using a single carrier, communicating in the near field. The ability to transmit power as well as data in the near field enables remote electronic circuits to be powered via the coupled carrier frequency and hence remove the need for a battery cell. This is important, for it allows circuits to be realized which are unencumbered with batteries and oscillators, providing the benefits of small size and improved reliability

The use of integrated circuits containing an identification number, which may be powered and interrogated by an inductively coupled field, is known as radio frequency identification or RFID. RFID systems are used to track objects which have been tagged, e.g. criminals, animals or assets in a supply chain etc. The programming and retrieval of identity codes requires a wireless communication with the integrated circuit (commonly referred to as a tag) and such interrogation is achieved using a scanning device called a reader. Data is subsequently stored in a database.

While useful, current RFID systems have a number of limitations the most important being the fact that they are functionally passive, i.e. they provide access to identification codes and other data etc. but are not active in terms of sensing or interacting within the tag's environment. Herein is described the development of an RFID type technology which allows the tag to both sense it's environment and affect change within that environment by means of actuation.

The motivation for this work concerned the desire to measure parameters of interest from the piston of an internal combustion engine when under load. This application presents a hostile environment for electronics, incompatible with battery technology and conventional integrated circuit fabrication technologies, both due to high temperatures of approximately 200 degrees Celcius. In addition the application limits the size and weight of the instrumentation, the opportunities for mounting the instrumentation and, due to reciprocating components, dictates the need for wireless technology.

The most important benefit which such an embedded instrument fulfils is that of measurement integrity. Normally, engine temperatures are sampled from accessible points around the engine, such as the oil sump temperature. This measurement however presents but an average



measure of engine temperature; it is impossible to distinguish whether one piston is running hotter than another from such a measurement. Therefore measuring such a parameter to increasing degrees of accuracy is unproductive, indeed measuring data from the most appropriate point in a system, even to a lesser degree of accuracy, is usually beneficial.

Another application concerns the embedding of the tele-sensing nodes into gypsum base dry-wall systems. The paper will also describe the use of such devices for smart-building earthquake and environmental monitoring.

The first section of the paper describes the development of a system capable of realizing a near field piston telemetry system compatible with extreme environments; culminating in the design of a robust, high temperature, radiation resistant integrated circuit; which has subsequently found use in other extreme environment monitoring applications. The second section describes how the extreme environment telemetry technology was extended to create a unique physical security and sensing system, capable of combating many of the threats associated with the misappropriation of materials from the bio-chemical, nuclear and military industries. In addition this system offers general consignment protection, tracking, anti-tamper and conformational change monitoring.

## SIGMA DELTA MODULATOR INTERFACES FOR MEMS CAPACITIVE SENSORS

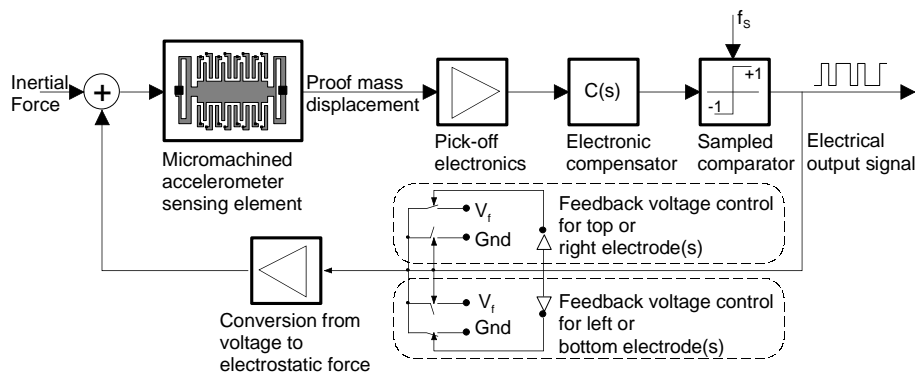
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Micro-electro-mechanical systems (MEMS) capacitive sensors, such as pressure sensors, accelerometers and gyroscopes have been one of the most successful examples of micro-system technology. They are routinely used in many commercial applications; including automotive safety systems, platform stabilization for video cameras, vibration monitoring, and many others. These sensors are typically open-loop and have low to medium performance specifications. Recently, there has been an emerging trend requiring more high performance sensors for more advanced automotive applications, inertial navigation and guidance, seismology, space sciences and many others. To fulfil this requirement it appears a very promising approach to apply some more advanced control and electronic interface techniques to existing micromachined sensing elements.

Incorporation of a capacitive sensing element in a closed loop, force-feedback loop can substantially improve the performance of these sensors, especially concerning their linearity, dynamic range and bandwidth. An ideal choice for the control system strategy is to use an architecture based on a sigma-delta modulator ( $\Sigma\Delta$ ). In such an approach the micromachined sensing element acts as the loop filter to form an electromechanical  $\Sigma\Delta$ . This type of control system yields a digital, pulse density modulated output signal that can directly interface to a standard digital signal processor. A typical example of such a sensing system for a MEMS accelerometer will be reviewed in detailed and their operation described. Fig. 1 shows a block diagram of such a MEMS sensor.



*Fig. 1. A closed loop electromechanical sigma-delta control system for a micromachined accelerometer.*

In such a system the sensing element solely acts as the loop filter, and provides noise-shaping only up to order two since the mechanical transfer function of the sensing element is of order two (mass-damper-spring system). It is well known from electronic  $\Sigma\Delta$  analogue to digital converters that second order  $\Sigma\Delta$  have some inherent problems; these include idle tones in the base band (limit cycle behaviour) and relatively poor noise shaping, especially at low to

medium sampling frequencies. Furthermore, for an electromechanical  $\Sigma\Delta$ M, the overall system performance is a strong function of the properties of the micromachined sensing element, and these are prone to considerable fabrication tolerances.

The most promising approach to overcome these shortcomings is to consider some higher order architectures routinely used for analogue to digital  $\Sigma\Delta$ M converters and to adapt them to MEMS sensors. It is necessary to add additional electronic integrators in series to the sensing element. A generic block diagram of such a system is shown in fig. 2. Several researchers have recently worked on this approach, including the authors, with very promising results. The approach, however, is not without challenge, especially stability and optimization pose a considerable challenge. The main problem regarding stability is that there is no possibility to have a local feedback loop tapping between the two mechanical integrators of the micromachined sensing element. The node between the integrators represents the velocity of the proof mass which is not accessible. A careful optimization process of the various gain constants is therefore required.

The seminar will discuss several higher order  $\Sigma\Delta$ M architectures, outline a design methodology resulting in stable systems and present a theoretical framework for analysis and parameter optimization. A case study of a fifth order electromechanical control system for a MEMS accelerometer will be presented, underpinned with simulation results and measurement data on a prototype implementation. As a second example, the approach will be discussed for a micromachined gyroscope. In this case, a band-pass  $\Sigma\Delta$ M control system is advantageous as the mechanical sensing element of a gyroscope can be treated as a mechanical resonator.

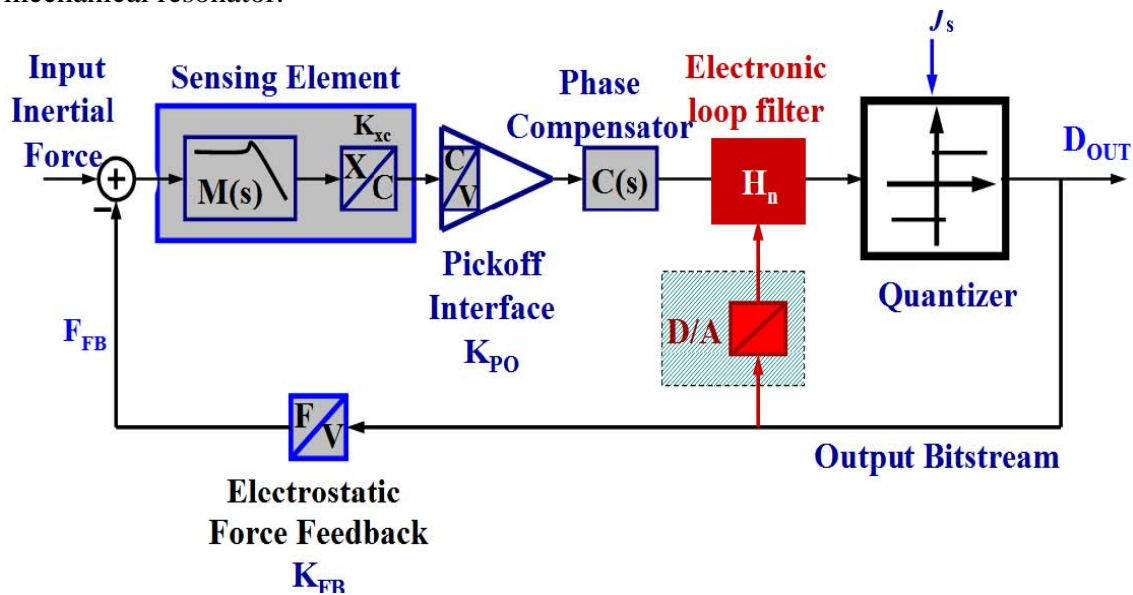


Fig.2 Generic block diagram of a high-order electromechanical  $\Sigma\Delta$ M sensor system. Cascading several electronic integrators with the second order mechanical transfer function of the sensing element forms a high order  $\Sigma\Delta$ M control system.

## DEFECT CHEMISTRY OF SENSOR MATERIALS

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Today's society is increasingly focused on automation and to be able to create a fully integrated system the existence of sensors is indispensable. A vast amount of different sensor systems is already in use today. These systems detect a broad spectrum of different parameters, e.g. light intensity, wave length, force, acceleration, flow, temperature, voltage, current, concentration of liquid or gaseous species, etc. This broad spectrum goes along with a variety of detection principles. A general definition is that "a sensor is a system that transforms a chemical or physical quantity into another domain, usually an electrical quantity".

In this lecture the focus will be on chemical detection systems and recent developments will be reviewed. While chemical detection systems comprise only a small part of the total sensor field, there is a large number of different chemical sensors on the market today. The differences are based on the medium in which they are applied, i.e., gas, liquid, or solid phase, the detection principle used, the operating temperature, and naturally the compound to be detected.

Here, we will discuss selected chemical and electrochemical sensors for a variety of gases, i.e., O<sub>2</sub>, H<sub>2</sub>, CO<sub>x</sub> (x=1,2), and NO<sub>x</sub> (x=1,2) as examples. In addition, advanced inorganic electrodes for the determination of environmental pollutants in surface waters will be presented. The focus will be on functional materials that comprise the sensors and electrodes, i.e., the ionically and mixed ionic-electronic conducting materials, and semiconducting materials. Hence, the relations between the structure and the defect structure of the functional materials will be addressed in relation with the responses of selected gas and electrochemical sensors. In order to discuss these relations, the principles of the defect chemistry will be presented along with the Kröger-Vink Defect Chemical notation. Furthermore, the role of nano-scale dimensions of sensor materials will be exemplified.

As an introduction, the defect chemistry of the solid electrolyte yttria-stabilised zirconia-based and niobium-doped titania-based Nernst-type, respectively, Taguchi-type oxygen sensors will be presented. Since the discovery of Iwahara and his group in the early 1980s, that rare earth-doped perovskites, adopting the perovskite structure, exhibit proton conductivity at elevated temperatures, these high-temperature ceramic proton-conducting (HTPC) materials have been studied in detail for applications such as hydrogen production from fossil fuels, hydrogen separation, hydrogen sensors, and proton-conducting fuel cells. Depending on the partial pressures of hydrogen, oxygen, and steam the electrolytic domain of, for example, SrCe<sub>0.95</sub>Yb<sub>0.05</sub>O<sub>3-x</sub> at 700°C will be discussed in relation to the defects in this cerate, i.e., electron holes, oxygen ion vacancies, OH lattice ions with an effective charge, and electrons, in relation to a hydrogen sensor application. The recent material

developments for hydrogen sensors will be presented in relation to their defect chemistries. Based on the proton-conducting solid electrolyte,  $\text{CaZr}_{0.9}\text{In}_{0.1}\text{O}_{3-x}$ , a novel catalytic asymmetrical Nernst-type sensor (CANS) has been designed for the indirect detection of methane via hydrogen detection. A room-temperature hydrogen sensor has been made using an array of titania nanotubes. Due to the photocatalytic properties of titania, the contaminated sensor is self-cleaning under UV irradiation.

Ti-doped chromium oxide (CTO) is being used as the active material for sensors to detect trace quantities of reducing gases in air, such as carbon monoxide and ethanol vapor. The defect chemistry of CTO has recently been reported and will be discussed. A new type of NO gas sensor was fabricated by the combination of aluminum cation and oxide anion-conducting solid electrolytes with lithium nitrate doped solid solution of gadolinium oxide-lanthanum oxide as the auxiliary sensing electrode. Here the solid-state and defect chemistry will be analysed.

Finally, copper-copper oxide, cobalt-doped lead dioxide, and graphite-based composite electrodes for the electrochemical determination of organic pollutants in aqueous environments will be presented.

# ***BOOK OF ABSTRACTS***

## ***3. Oral Contributions***



# THE ELASTIC HALF-PLANE WITH A THIN INHOMOGENEOUS COATING UNDER THE ACTION OF A STRETCHING FORCE APPLIED AT INFINITY

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The materials used nowadays in health industry, chemistry and biochemistry have a complicated structure, as coatings and layers allow changing the characteristics of the material and increase its possibilities to resist heat, wear, chemically aggressive substances, etc. As new composite materials appear, very often the solutions of classical problems can't give any more adequate answers about their behavior under the different mechanical influences. So new problems must be solved as theoretical results and good mathematical models usually reduce the number of necessary expensive experiments.

In the present work the problem of the distribution of the contact stresses between the elastic half-plane and a rigid sensor (of finite dimensions) is considered. The half-plane, having a thin coating inhomogeneous in depth, is exposed under the action of an external force applied at  $+\infty$  and  $-\infty$  parallel to the surface. The solution of the problem in its classical formulation (in case of a half-plane without a coating) is well-known and considered in detail in the work by Alexandrov V. M., Mkhitaryan S. M. "The contact problems for solids with thin coatings and layers", M., Nauka, 1983, 488 p. For the new formulation of the problem dual integral equations for displacements directly under the sensor are obtained. The numerical approximations of the Fourier Transforms of the displacements allow finding the contact stresses.

Originally this problem appeared because of using sensors for gathering information about the material. The developed model may be used in systems of security or tracking since it allows determining stresses inside a material with inhomogeneous or graded coating considering the dimension of stretching stresses brought by a covering layer. The distribution of contact and shift stresses under a sensor let us judge whether the investigated object is homogeneous or it has a complex structure representing a package of layers or graded materials.



## MESOPOROUS INDIUM OXIDE FOR GAS SENSOR APPLICATIONS

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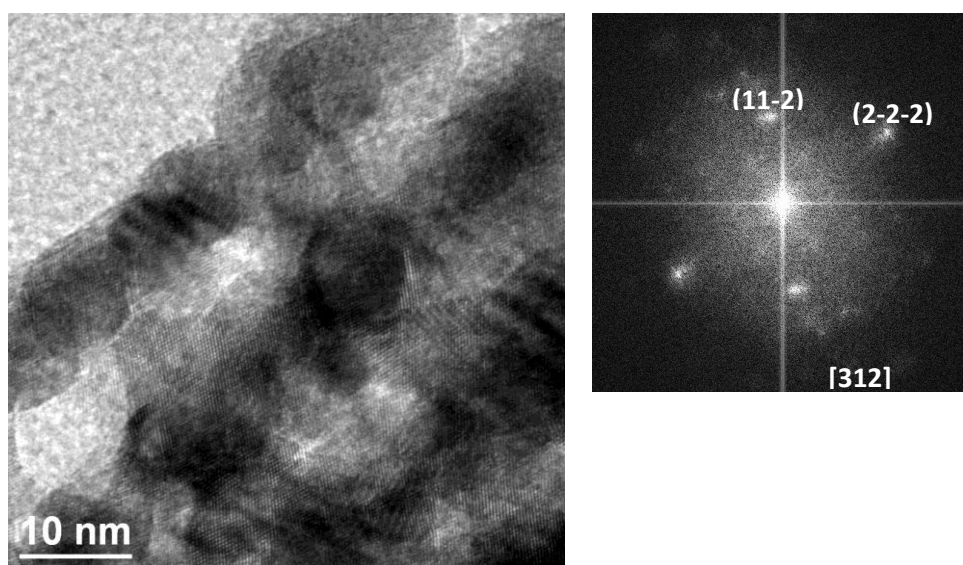
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Nanostructured mesoporous materials have been widely studied in the development of novel material performances, due to their large, controllable pore size and high surface area that make them outstanding for applications that require gas interactions. On the other hand,  $\text{In}_2\text{O}_3$ , due to its surface reactivity characteristics, is a material widely used for the detection of reducing or oxidant gases, such as carbon monoxide or nitrous oxide, important for monitoring environmental pollution resulting from combustion or automotive emissions.

In this work, we have used mesoporous silica SBA-15 (two-dimensional hexagonal structure) and KIT-6 (three-dimensional cubic structure) as a template for the synthesis of different  $\text{In}_2\text{O}_3$  mesostructures. Both silica templates were synthesized by a sol-gel route, in aqueous acidic conditions using Pluronic P123 triblock copolymer (EO20PO70EO20) as a structure promoter and tetraethyl orthosilicate (TEOS) as silicon source; in the case of KIT-6, 1-butanol was also added.

For the synthesis of mesoporous  $\text{In}_2\text{O}_3$ , a two-impregnation step was used. An amount of silica template (SBA-15 or KIT-6) was suspended and stirred in ethanol with the oxide precursor,  $\text{In}(\text{NO}_3)_3 \cdot x\text{H}_2\text{O}$ . The mixture was dried and calcined for 4h at 350°C for the first impregnation and 550°C in the second impregnation. Finally, the template was removed using 2M NaOH at 70°C for 24h. The obtained materials show small particle sizes, about 5-10nm, and high surface area and their performances have also been compared with quasi-spherical nanoparticles of similar size. Sensors have been prepared on alumina and micro mechanized hot plates. Their structural and electric characterization will be reported and discussed.



BFSTEM image and FFT of nanostructured  $\text{In}_2\text{O}_3$  grown in a KIT-6 silica template

# CORRELATION ANALYSIS OF THE NOISE SENSOR SIGNALS BY THE ADAPTIVE PROCESSOR OPERATING USING A NUCLEAR SPIN ECHO PHENOMENON

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The sensory operation, in particular, the broadband signal processing and correlation analysis is a quite intricate problem since it requires fast adaptation of a device to the signal characteristics. In our experiments with echo signals obtained from the ferromagnetic or superconducting materials, this problem can easily be solved. We can register the interrelation of noise signals received from different sensors in a real time, and as a result, to eliminate the sensor intrinsic noises. It is also possible to determine the normalization start time in the noise process registered by one sensor. The last effect can lead to irreversible changes in the object controlled by the sensor.

The application of NMR technique for a storage of the information in quantum computers as well as using a nuclear spin echo phenomenon to reproduce this information were discussed by authors in [1], where noise signals of various duration were considered as a model of random numbers. The echo can be used for certain information processing too (see review [2]) including its application in the Data Acquisition Systems to collect signals from primary sensors. In the present paper we describe experiments on excitation of an echo by pulses with noise and harmonic filling in thin cobalt films.

In the theoretical consideration, the echo signals obtained are interpreted as a correlation function of the noise excitation pulses. If these pulses are considered as a realization of some random sequence or function, the arisen echo can represent a convolution operation of these sequences (functions). The experiments with noise signals show that the echo phenomenon can easily be adapted to processing of various kind of the information coming from sensors of any type.

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## SYNTHESIS AND EXAMINATION OF HEXAGONAL TUNGSTEN OXIDE NANOCRYSTALS FOR ELECTROCHROMIC AND SENSING APPLICATIONS

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Tungsten oxides and tungsten oxide hydrates are among the most used materials in electro-, photo- and gasochromic applications. Lately, tungsten oxides are commonly applied as sensing layers for hazardous gas detection as well. In this work, a soft chemical nanocrystalline processing route has been demonstrated for the preparation of hexagonal tungsten oxides. The acidic precipitation of Na<sub>2</sub>WO<sub>4</sub>·2H<sub>2</sub>O solution was followed by hydrothermal treatment of the resulted tungstic acid hydrate gel and then the dehydration of the oxide-hydrate at temperatures as low as 120 °C and 330 °C. The morphology of parent phases, such as layer structured rectangular crystallites of WO<sub>3</sub>·H<sub>2</sub>O, orthorhombic WO<sub>3</sub>·1/3H<sub>2</sub>O, and resulting oxides with open structured nanosize hexagonal platelets and rods of h-WO<sub>3</sub> particles have been studied by scanning electron microscopy. Structural and electrochemical performance of thin films have been determined by SEM, TEM, XRD, Raman spectroscopy, atomic force microscopy and cyclic voltammetry. The ion insertion properties of tungsten oxide hydrate and tungsten oxide films show a clear dependence to the presence of structural water and to the close packed structure. Sensing properties of the prepared tungsten oxides have been tested to gaseous ammonia at various temperatures. Sensor films showed a decrease in resistance on exposure to NH<sub>3</sub> as is characteristic of an n-type semiconductor. The layers were found to be sensitive in the concentration range of 50-500 ppm at the temperature of 300 °C.

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**METAL OXIDE SILICON BASED AVANCED SENSORS****Arezki BENFDILA**

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The present paper deals with the advances in sensor systems based on the Metal Oxide Semiconductor Field Effect Transistor (MOSFET) widely used today in a variety of applications. MOS sensors based on ZnO and SnO<sub>2</sub> are used since 1960s as detectors for liquid petroleum gases (LPG) in homes. Due to their successful operation, they are explored further for various other applications ranging from civil to military. In addition to the sensors prepared from oxides as abovementioned, other sensors that are currently produced in large quantities include oxides, such as TiO<sub>2</sub>, WO<sub>3</sub>, In<sub>2</sub>O<sub>3</sub> and other similar oxides.

At the present time, applications of MOS detectors vary widely across scientific disciplines to include RF, nuclear, chemical, light, microwave etc. Such detectors vary in dimensions from a fraction of a micron to several thousand microns and can be used for several applications ranging from chemical to biological detections. The present communication consists of the state of the art in MOS advanced technologies and the techniques used to design and manufacture high performance detectors and sensors for different applications. Examples of chemical, light and microwave detectors will be presented. In addition, application of MOS devices for image sensors and medical sensors will also be described. Emphasis will also be placed on the microdetectors that are embedded in integrated circuits and systems. Furthermore, systems and device operations are described in conjunction to the sensor purpose. The engineering and fabrication aspects in conjunction with the material properties and characteristics are discussed. One of the key features of such MOS detectors is convergence of large to medium scale integration for development of such hybrid circuits.

**Keywords:** MOS system, Sensors, Chemical sensing, Microdetector

## MICROSENSORS BASED ON NANOSIZED STRUCTURES OF PRUSSIAN BLUE FOR HYDOGEN PEROXIDE DETECTION

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Electrode with electrochemically deposited Prussian Blue on the surface was shown to be an advanced electrochemical sensor for hydrogen peroxide detection [1]. Application of microelectrodes allows to increase the sensitivity of analysis and to decrease the detection limit with respect to conventional electrodes.

Microelectrodes have been prepared using gold wire (125  $\mu\text{m}$  diameter) and medical catheters. Alternatively, microelectrodes have been made from common single-core cable (100  $\mu\text{m}$ ). Cable armature has been covered with silver paste and then electrochemically modified with AgCl. Working electrode surface has been modified with carbon paste, or Ni layer with following Au layer deposition. Finally electrochemical deposition of Prussian Blue has been done.

Hydrogen peroxide microsensors possessed unique analytical characteristics. Accordingly, microsensor system in flow-injection analysis revealed the detection limit as low as 10 nM  $\text{H}_2\text{O}_2$ . Linear calibration range prolonged from 10 nM to 100  $\mu\text{M}$   $\text{H}_2\text{O}_2$ . Total response time was about 30-60 s.

AFM investigation confirms the polycrystalline structure of Prussian Blue film deposited on electrode surface. The size of crystalline was of about 50-100 nm. Average thickness of Prussian Blue film was estimated to be about 60-70 nm.

The developed system based on integrated microsensor for hydrogen peroxide is highly attractive for applications in modern clinical diagnostics.

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## DONORS IN SEMICONDUCTORS : THE PAST OR THE FUTURE OF SEMICONDUCTOR ELECTRONICS

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The physics of semiconductors, and, consequently, contemporary electronics, cannot be understood without impurities, as has been recognized in early experimental and theoretical studies of semiconductors in years 1930-1940. The hydrogen-like shallow donor (and acceptor) state of electron (hole) bound by Coulomb electrostatic force of excess charge of impurity was at the base for constructing semiconductor diodes, transistors and numerous types of semiconductor electronic and optoelectronic devices, including lasers and detectors, in the following decades.

Recently, surprisingly, the physics of impurity donors appeared to be much richer. Numerous experimental evidences have been provided for universal existence of other types of electronic states of the same donor impurity:

- i) mysterious, deep, DX-type states resulting in metastability - slow hysteresis phenomena - understood as two-electron, acceptor-like states of impurities, formed upon large lattice distortion or rearrangement around impurity, resulting in negative electron correlation energy  $U$ ;
- ii) deep, localized, fully symmetric, one-electron donor states of substitutional impurities.

The latter state can be formed from the "ordinary" shallow hydrogen-like state in the process of strong localization of electron by short range, local potential of impurity core, preserving full ( $A_1$ ) symmetry of the substitutional impurity in the host lattice. The "anti-crossing" of energy levels upon transformation is observed.

All types of electronic states of impurity can be universally observed for the same donor impurity and mutual transformation between different states can be observed upon changing experimental conditions.

The knowledge about existence and properties of these "new", molecular type, donor states in semiconductors seems still await general recognition and positive application in contemporary material and device science and engineering.

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## BASIC DEVELOPMENT OF FLOW IMMUNOSENSORS FOR ORGANIC POLLUTANTS

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The development of different immunosensing systems for the determination of organic pollutants is presented herein. The systems make use of the competition between the analyte and a labelled hapten for the binding sites of a specific antibody. This process takes place in solution, and the formed immunocomplexes are further captured by immobilized protein A/G. The activity of the label bound to the capture support is related to the analyte concentration in sample. Capture immunosensors have been applied to insecticide residues (carbaryl, 1-naphthol, 3,5,6-trichloro-2-pyridinol), herbicides (atrazine, glyphosate), antifouling agents (irgarol 1051) and antibiotics (sulfathiazole). Analytes can be determined at the µg/L level, and even lower, with a total assay time of 18 minutes per sample, that can be accelerated till 8.5 minutes although sensitivity is partially lost. The possibility of employing the sensors with organic solvent extracts has been studied and methanolic extracts diluted 50/50 (v/v) with buffer has been analyzed. The immunosensing systems have been applied to different matrixes such as water -drinking, river, sea-, food and soil, with good results.

## MAGNETIC IRON OXIDES AS POSSIBLE SENSORS FOR TOXIC ANIONS IN WATER STREAMS

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Toxic anions (i.e. oxyanions of As, Cr, Se, etc.) present an increasing concern today as they are present in water streams from natural as well as anthropogenic sources. Among them, As is considered the most toxic and has been identified as a high priority pollutant, both in EU and US. The well known As related health problem of Bangladesh made worldwide headlines and resulted in the decrease of maximum As limit in drinking water to 10 µg/L.

The water supply systems of large cities can handle quite well the removal of moderate concentrations of As from water streams. However, in developing countries and in small water supply systems in developed countries, the removal of As to levels below the limit is problematic. Even the good installations of large cities can not handle well increased concentrations of As, which could be put there on purpose (i.e. terrorist attack). Consequently, the development of sensors for rapid detection of As contamination in water streams is of paramount significance.

A good sensor should have an easy measurable property, should react quickly with increased concentrations of the detected substance and give the appropriate signal promptly. Magnetic iron oxides are considered as good candidates for sensing As oxyanions in water streams. They have good magnetic properties, they react with arsenates and can be produced in nanoparticles, which will increase substantially the surface area and consequently the kinetics of the process to levels acceptable for use as sensors.

In the present research, synthetic magnetite nanoparticles have been produced by co-precipitation of Fe(II) and Fe(III) in the presence of NH<sub>4</sub>OH, at temperature 70°C. Their mean size has been determined to 20-50 nm. The magnetite particles have been tested for As removal and the process was found to be quite fast. Thermodynamic analysis of the magnetite-As-water system as a function of pH and Eh have shown that scorodite (iron arsenate) is the thermodynamically stable phase when arsenates are sorbed on magnetite surface. Hence, a change of the magnetic properties is expected. Systematic analysis of magnetite samples before and after arsenate sorption will determine the extend of scorodite formation and the difference in the magnetic properties of the material.

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# RESPONSE OF NERVE CELL TO INHIBITORS RECORDED BY AlGaN/GaN FIELD EFFECT TRANSISTORS

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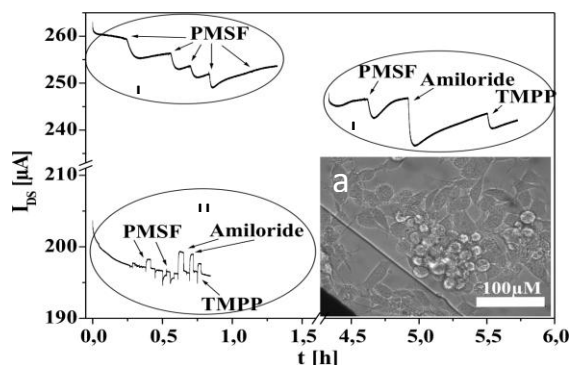
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In this work we report on the microscopic recording of the extracellular potential of NG108 – 15 (mouse neuroblastoma x rat glioma hybrid) nerve cells as response to three different inhibitors using an open gate AlGaN/GaN electrolyte gate field effect transistor (EGFET) [1, 2]. Sensing of gases, ions, (bio-) molecules and the determination of the pH-value in solutions using AlGaN/GaN FET have been recently demonstrated [3]. The sensor chip was encapsulated into a specially created well plate which can be very easy converted into a measurement setup with an integrated Ag/AgCl reference electrode.

The non - metalized gate surface of our sensor was not functionalized by organic materials. Previous biocompatibility studies of our GaN materials with NG 108 – 15 living cells showed a proliferation rate of about 95%. The PMSF (phenylmethanesulfonylfluoride), Amiloride and TMPP inhibitors were added to the medium with and without adherent cells (NG108 – 15) and the source-drain current ( $I_{DS}$ ) of the AlGaN/GaN field effect transistor was recorded versus time (Fig. 1). Using AlGaN/GaN EGFET, we observed the extracellular activity of the same cell field over long time and our device has shown stable operation under physiological conditions and a very good signal resolution S/N ratio of  $> 30:1$ . The cells react very different to the specific inhibitors in the case of repeated titration of the PMSF inhibitor a saturation concentration was determined, where no further cell reaction was detectable. The observed changes in  $I_{DS}$  are affected by chemical recombination, and the enhancement of charges in the thin electrolyte layer between the cell membranes and the semiconductor surface. Consequently AlGaN/GaN electrolyte gate field effect transistors are highly suited for the detection of the cell electrical signature on a micro scale as response to modulation of the ionic channel properties upon toxin expose.

FIG. 1.  $I_{DS}$  versus  $t$  recording of medium, with (I) and without cells (II), reaction to different inhibitors.  $V_{DS} = 0.5$  V and  $V_{GS} = -0.65$  V were kept constant. Insert a) shows NG 108-15 cells cultivated on the device surface.



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## DEVELOPMENT OF BIOLOGICAL SENSORS BASED ON SCREEN-PRINTED ELECTRODES FOR ENVIRONMENTAL POLLUTION MONITORING

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In the last years, an increasing interest has been observed for control and monitoring of food and environment quality and safety. The biosensors seem to be a good alternative to the conventional methods for this purpose, as they can provide fast and specific data, allowing the detection of a broad spectrum of analytes in complex matrices.

Screen-printed electrodes (2 and respectively 3 electrodes configuration) modified with Meldola's Blue mediator have been used for the detection of dithiocarbamate fungicides. Metham-sodium is a potential alternative to methyl bromide, used as broad spectrum soil fumigant, with no effect on the stratospheric ozone layer, but its degradation leads to methyl isothiocyanate (MITC) and hydrogen sulfide, which are moderately to strongly poisonous.

Biosensors for the detection of metham-sodium and MITC were designed in two configurations, either by coupling aldehyde dehydrogenase with diaphorase or using only aldehyde dehydrogenase, the enzymes being entrapped in both cases in PVA-SbQ polymer. It was shown that, contrary to other dithiocarbamates, metham-sodium did not inhibit ALDH, while MITC could be detected at levels of 100 ppb. The developed sensor allowed thus discriminating between metham-sodium and its toxic metabolite MITC.

Now, our work is devoted to the development of screen-printed electrodes modified with single-walled carbon nanotubes and a proper redox mediator for sensitive detection of alkyl phenols (APs) and their derivatives alkylphenol ethoxylates (APEs), which are mainly used as stabilizers for rubbers and plastics, as surfactants, as industrial detergents, and in the mining and textile industries. Alkyl phenols are of increasing concern due to their chemical functional similarity to estrogen and other hormones, binding to the estrogen receptor and inducing certain estrogen-specific biological responses *in vitro* [1-3].

The remarkable properties of single-walled carbon nanotubes (SWNTs) have attracted and increased the interest for a broad spectrum of sciences and technologies including chemistry, biology and medicine [4].

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## MULTI-FUNCTIONAL NANOMATERIALS FOR ADVANCED BIOLOGICAL/CHEMICAL SENSOR TECHNOLOGIES

**Aisha BISHOP-HAYNES**<sup>1,2\*</sup> and **Perena GOUMA**<sup>2</sup>

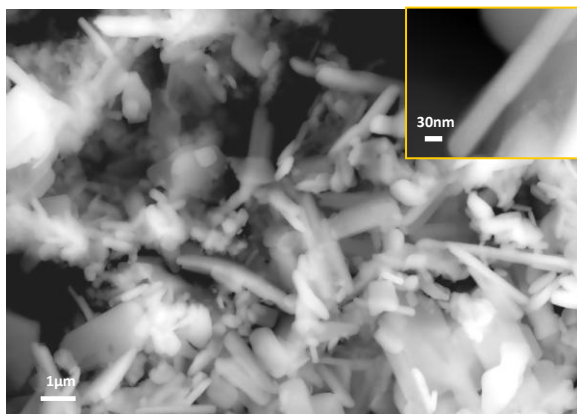
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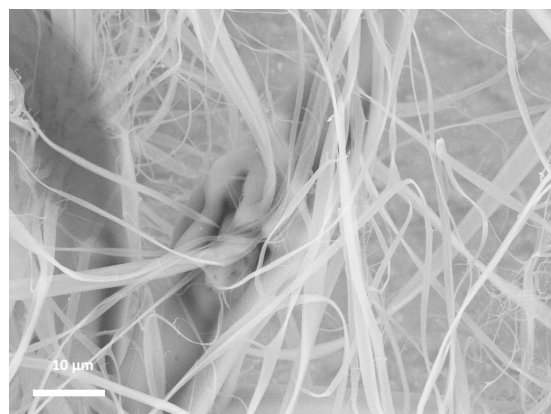
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This research focuses on the science and development of single crystalline metal oxide nanowires and polyaniline nanofibers constructed using the electrospinning technique. The novelty of this research is the production of mono-phase, single crystal metal oxide based materials, figure 1, and active organic matrices based on reduced polyaniline, figure 2. The electrospinning technique provides an added advantage in that it offers single step processing of these nanoarchitectures yielding a controlled “top-down” processing mechanism for development of nanowires and nanofibers that exhibit high sensitivity and specificity to target analytes in varying environmental conditions.

These novel sensor membranes have never been investigated, especially for defense and security applications. One product of this research is an advanced array of real time metal-oxide nano probes and polyaniline hybrid nanofibers (for use in personal monitoring technologies) which can selectively detect varying chemical and/or biological agents.



*Figure 1: Scanning electron microscopy image of the nanowire mat produced after calcinations of electropsun metal oxide composite; (inset) High magnification image of a single MoO<sub>3</sub> nanowire*



*Figure 2: Scanning electron microscopy image of electrospun cellulose acetate-lecuoemeraldine based polyaniline composite fibers*

## DETERMINATION OF THE MUTAGENIC EFFECTS OF POLLUTION BY AMES AND NEURAL NETWORKS

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This study presents a novel artificial neural networks approach on determination of the mutagenic effects of pollution. Considering those points, water samples are taken from 20 different locations in Bosphorus. Those samples are lyophilized by using an evaporator. Lyophilized samples are dissolved in three different organic solvents to make them applicable for the AMES (*Salmonella*/Microsome Mutagenicity Test System). For this purpose, a multi layer perceptron (MLP) architecture which consists of Back-Propagation training algorithm was used. The five inputs of MLP are normalized value of average, standard deviation, middle value, and standard error, and T value (results of comparing) of the number of *Salmonella* bacteria strain, TA-98. The output of MLP is classification of mutagenic, non- mutegenic, and weak mutagenic situations. As it can be seen the monitoring of ANN software program in Figure below was developed by using Pascal under Delphi. The results of proposed study show that samples collected from different locations in Istanbul.

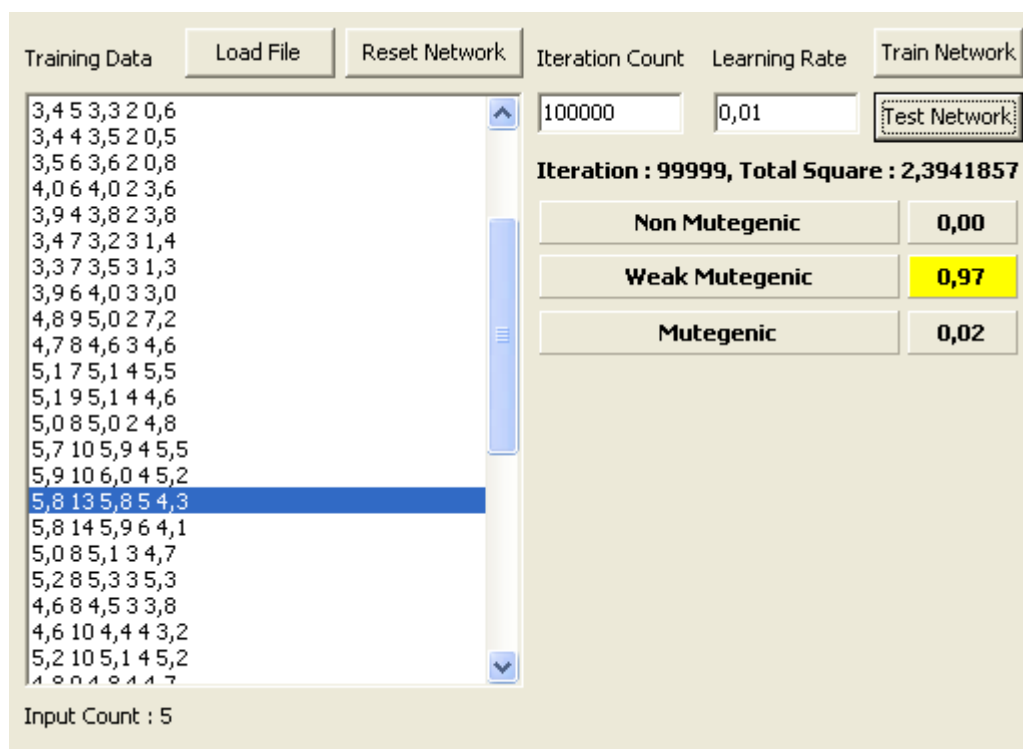


Figure: The program monitor

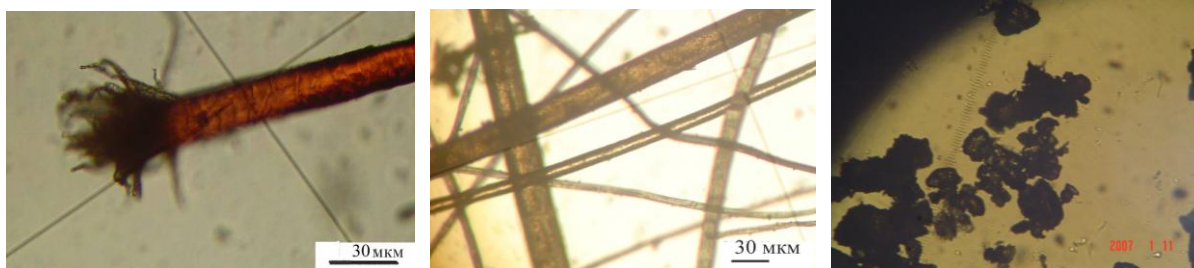
## NOVEL MOLECULAR CRYSTALS OF CARBON AS NEW PERSPECTIVE ELEMENTS OF NANOSENSORS

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One-dimensional structures are the most perspective objects of miniaturization of elements of nanoelectronics, and in particular, nanosensors. Carbon nanostructures already find practical application because of brightly expressed dimensional effects. Here we represent original experimental results connected to obtaining of new structures of transparent carbon. Let's remind that two of three known allotropic modifications of carbon - diamond and fullerene - are transparent and both have cubic crystal structure. If the diamond is formed at superhigh pressure and increased temperatures, fullerenes are formed in conditions of extremely high gradients as on temperature and pressure. We obtained 2 new kinds of crystals of transparent carbon at perfectly new reactionary conditions. The images of optical microscopy (in transparent polarized light) of new molecular crystals of carbon are presented in figure.



The transparent thread-like carbon crystals painted in various colors are formed at high-thermal exposure powders of carbon and silicon. From a product containing nanothread-like SiC, the painted threads are found out by a diameter up to 40 microns and length more 30mm and are allocated by means of optical microscopy. According to crystalloptical analysis these thread-like crystals are anisotropic with a parameter of two-refraction close to quartz. However according to X-ray analysis threads have original, distinct from quartz, hexagonal crystalline structure with the following parameters:  $a = 0,498\text{nm}$  and  $c = 0,826\text{nm}$ . The chemical composition of threads was appreciated with the help X-ray spectral analyzer (Camebax). Basic element of threads is the carbon. Silicon and oxygen contain in very small amounts. Study threads by means of microanalyzer (Superprobe-733) also have shown, that carbon contains in all investigated samples, whereas silicon in some threads is not revealed at all. The distinctive feature of carbon threads is, that they are formed from great number of threads of a smaller diameter and consequently have the specific ending (see pictures above).

Isotropic particles (look pictures above) of transparent carbon are precipitated from benzene solution, which is obtained at pan out by benzene of pyrolytic soot. Pyrolysis of aromatic hydrocarbons is carried out in reactionary conditions similar to synthesis of carbon nanotubes. Isotropic particles of carbon are anisotropic crystals with a high parameter of two-refraction. As against fullerene new molecular crystals of carbon are characterized by high thermal stability (are synthesized at temperatures above  $1000^{\circ}\text{C}$ ) and stability to the solvents of a various chemical nature.

## SELECTIVE DETECTION OF TRACE EXPLOSIVES USING PIEZORESISTIVE MICROCANTILEVERS

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A widespread need exists for developing trace explosive sensors with high sensitivity and selectivity to combat explosive-based terrorism. However, achieving high selectivity in trace explosive detection is a challenging task. Although there exist many sensitive platforms such as microcantilever sensors, they do not have any intrinsic selectivity. Chemical selectivity in detection is often achieved by using immobilized selective receptors on the cantilever surface. Receptor-based detection leads to increased false positives due to the lack of highly specific receptors. We have developed a cantilever sensor concept for trace explosive detection that does not utilize any receptors. In this technique explosive vapors are allowed to adsorb on the surface of a piezoresistive cantilever. The adsorbed explosive vapor on a cantilever is then made to interact with ozone, which is locally generated by the interaction of UV light with oxygen in air. The interaction of ozone with adsorbed explosives creates a characteristic mechanical response with respect to a reference cantilever. The mechanical signature of the cantilever is measured as changes in the resistance of the piezoresistive cantilever beam. The concept is amenable for miniaturization since consumable ozone is generated locally using a pencil-sized UV lamp. Results from detection of TNT, RDX, and PETN will be discussed.

## A ROADMAP FOR MATERIALS FOR SECURITY

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To identify innovation potentials for materials for a safe Europe, experts were interviewed and a roadmapping workshop was carried out within the “SMART” project.

SMART (“ForeSight Action for Knowledge-Based Multifunctional MAteRials Technology”) is a Specific Support Action funded by the European Commission within the 6<sup>th</sup> Framework Programme. The objective of this project is to provide both the scientific community and the European Commission with important information about the specific strengths and weaknesses of materials technologies in Europe as well as to evaluate perspectives for future materials research.

The roadmapping process consisted of four stages in which (1) the current situation, (2) the future direction, (3) the barriers to progress and (4) the recommended actions to overcome these barriers were identified and discussed by experts. In the talk, the findings of each stage will be outlined in order to gain a deeper understanding of the presented roadmap for materials for security. The identified topics connected to sensors and sensing materials are:

- Non-stop security checks for personal protection
- Biometric identification
- Anti-counterfeiting
- Tools for forensic sciences.

Further priority actions are listed as cross-sectorial issues in the roadmap. Some of the technology developments identified and future actions are described in the subsequent paragraphs.

Nature has numerous examples of extremely highly sensitive sensors. The rapid and accurate determination of low concentrations of toxic agents and biohazards can be achieved by nanotechnology and lab-on-a-chip techniques as well as biological sensors. In the field of multi-spectral detection systems at terahertz frequencies, more basic research on appropriate radiation sources is required. Self-healing and self-checking detection systems are generally also required and standards for sensors should be established.

Smart materials play an important role in the development of security systems. Examples for the security-related use of RFID are applications in passports for authentication purposes and in consumer products to hinder counterfeiting. For future progress in this area, the development of advanced polymers for printed electronics has been identified as a critical research topic. In spite of the aim to improve security for the European citizen, privacy aspects must also be taken into account. In order to raise the public acceptance of security measures, the benefits of sensors should be promoted.

Materials research can pave the way for a wider application of sensor technologies through the development of advanced sensor materials for detection systems with increased sensitivities and selectivities as well as for ease of manufacturing.

## POROUS SILICON BIO-CHEMICAL SENSORS

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Porous silicon is a unique and versatile material that has several features that make it especially attractive for biological and chemical sensors, including a very high surface area to volume ratio, simple and inexpensive fabrication techniques, and suitability for integration with silicon electronics.

Offered architecture and method of making bio-chemical sensors provide a sensitive way to measure small changes in the in electrical properties (capacitance and conductance) of porous silicon that occur when exposed to organic solvents or when biological molecules attach to the internal surfaces. In particular, use of unilateral electrical contact allows a complete exposure of the surface to the sensing species and reduces the generation of ionic currents through the porous matrix. Pore selective distribution (gradient in pore sizes) in matrix increases sensitivity of sensors.

Based on offered sensor engineering prototype of portable, hand-held diagnostic device is developed. Such sensitive label-free devices can be used by consumers worldwide, for example to detect the presence of specific biological agents (viral DNA, proteins, and potentially bacteria) or organic solvents (ethanol, acetone, benzene).

Preliminary testing demonstrated the main advantages of the proposed sensors, including miniaturization, portability, high throughput, high signal to noise ratio, and production of on-site and real-time results.



## NEW APPROACHES TO SYNTHESIS OF SENSING MATERIALS FOR THERMOCATALYTIC AND SEMICONDUCTING GAS SENSORS

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The aim of this research was to develop and realize synthetic techniques for preparation stable and selective sensing materials. For this, we performed a systematic study of the influence of synthesis method, matrix composition, and crystalline structure of gas sensing material on sensing properties. In this presentation we report on comparative analysis of physicochemical properties of two types initial materials: home-made SnO<sub>2</sub>, synthesized using advanced precipitation technique and commercial available SnO<sub>2</sub> nanopowder (Sigma Aldrich). We report on sensing properties of these different materials for hydrocarbons as well.

The general idea of the sensing material preparation is not to use compounds containing elements (e.g. Cl, S, Na, K) which hardly could be scavenged by means of traditional preparative chemistry. The best solution is provided with the use of metal-organic compounds. Thus, for synthesis pure tin dioxide we used tin (IV) acetate complex as precursor in conventional precipitation technique.

The XRD analysis showed rutile-type structure (cassiterite) for both synthesized and commercial oxides. Thermo XRD measurements allowed us to reveal crystallite ability to grow under heat treatment. By means of profile fitting software TOPAS and method Length Diffraction Column Volume Integral Breadth (LVol-IB) we calculated mean crystallite size of two materials at different temperatures (22, 300, 400, 500, 600, 700° C) and during isothermal heating at 700° C. It was found that initial growth temperature for both materials is the same in the frames of the errors of profile fitting ~ 400 – 500° C. The limiting crystallite size for synthesized SnO<sub>2</sub> after 10 hours of annealing at 700° C is ~ 6 nm, the crystallite size of commercial SnO<sub>2</sub> after the same procedure is 34 nm.

Laser spark element analysis showed that commercial SnO<sub>2</sub> contain a significant amount of Na and Cl ions – about 0.3 and 0.4 mass % of each element, respectively. And even after repeatedly washing in bidistilled water the quantity of the impurities did not decrease very much. Also for material analysis we used IR and Raman spectroscopy. It was shown that even after drying at 300° C (10 hours) synthesized SnO<sub>2</sub> contains significant quantity of adsorbed water than commercial one. But at the temperature 450° C material seems to be free of remarkable water content.

Gas sensitivity measurements to methane were carried out for doped SnO<sub>2</sub> with 1.5 w. % of Pd and 0.5 w. % of Pt and pure home-made SnO<sub>2</sub>. It was found that pure synthesized SnO<sub>2</sub> possesses high catalytic activity and exhibits the same signal to 0.6 vol. % of CH<sub>4</sub> in comparison with doped material on the basis of commercial tin dioxide. The doped home-made SnO<sub>2</sub> possesses extremely high sensitivity to the same gas, which is more than by 10 times higher than for commercial doped SnO<sub>2</sub>. The sensors signal defined as the quotient  $R_0/R_g$  for this methane concentration is equal 6.1, 6.3, 83.7 (pure home-made, doped home-made and doped commercial SnO<sub>2</sub>, respectively).

## THE MONITORING OF THE LAND SURFACE BIOMASS BY MEANS OF THE MULTI-ANGULAR SENSORS – A WAY TO ENHANCE THE ENVIRONMENTAL AND SOCIAL SECURITY

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The energy security is a very hot subject for the international community in our days. It is related sometimes with menaces of terrorist attacks and damages in infrastructures of the oil and gas productive countries. The energy security subject is based on several issues. One of the most important is that a biomass provides an efficient energy substitute for fossil fuels. Many countries demonstrated substantial progress in development of such technologies during last decade. This approach has another important advantage: biomass is a sink for sequestering carbon, which accelerate a greenhouse warming effect in Earth Climate System. Latter is of high priority concern issue for the international community as well as energy security one. Therefore, any advance in solution of above problems moves us towards more sustainable and predictable world. Growing and using biomass on a continuous basis as a substitute for fossil fuels has clear advantage compared to using the biomass solely as a means to sequester carbon to create carbon sink. Renewable-grown biomass is a carbon dioxide-neutral fuel with a low sulphur content and can be converted in electricity, heat, liquid and gaseous fuels. In addition, rural communities gain jobs rather than removing land from productive use only. Thus there are numerous environmental and social security advantages to be gained from growing and producing biomass energy. Solution of above problems is related to many particular subjects. Long term global scale land biomass monitoring is a key tool for biomass growing strategy international control. Therefore it is one of priority issues. Satellite sensors providing multi-angular reflectance data in global scale but with a high spatial resolution form the background of the Earth remote sensing observations for the new decade. The synoptic and frequent viewing capabilities of the new generation of multi-angular sensor systems provide a comprehensive means for a global land surface monitoring. Nevertheless, prior characterizing the physiological status of the vegetation it is mandatory that a special attention be given to the all pre-processing steps. Satellite measurements can provide a good sampling of the bi-directional reflectance distribution function of terrestrial targets that is further used now in many applications - climate modelling, hydrology, ecology, biology, and soil sciences. It remains that the retrieval of key surface properties still require to put some effort in searching stable solutions to the inverse problems of physically-based algorithms. Of particular interest are estimates of the vegetation biomass, and leaf area index. Biomass data can only be obtained at proxy for polar orbiting sensors like AVHRR. Such product is being superseded thanks to the advent of new multi-angular sensors such as: POLDER (Polarization and Directionality of Earth Reflectance), SeaWiFS (Sea-Viewing Wide-Field Sensor), VEGETATION, MISR (Multi-Angle Imaging Spectra-Radiometer), MODIS (Moderate Resolution Imaging Spectroradiometer), MSG (Meteosat Second Generation) and MERIS (Medium Resolution Imaging Spectrometer). The algorithms for the evaluation of the biomass data generated by these sensors are considered. Many illustrative examples attributed to various land cover types and geographic areas are presented as well. The estimations for the biomass monitoring accuracy bands are discussed.

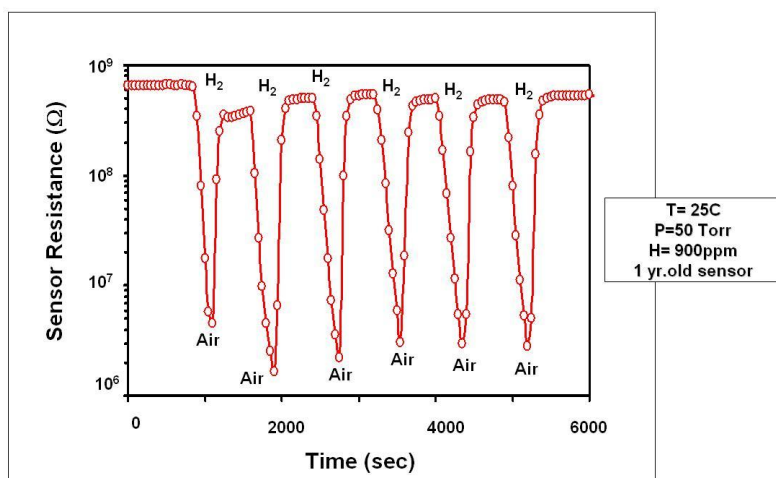
# NANO-CRYSTALLINE INDIUM DOPED TIN OXIDE GAS SENSOR

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MEMS based Indium doped Tin Oxide ( $\text{SnO}_2$ ) gas sensor device with sol gel synthesized mesoporous nanocrystalline ( $<10$  nm) semiconductor thin ( $100\sim150$  nm) film has been developed. The novel room temperature hydrogen sensor has a fast response time (less than 10s) and quick recovery time, as well as high sensitivity ( $R_a/R_g=10^4\text{-}10^5$ ). The improved capabilities are credited to the large surface to volume ratio of gas sensing thin film with nano sized porous surface topology, which can greatly increase the sensitivity even at low working temperature. Since the fabrication process is completely compatible with IC industry, it makes mass production possible and greatly reduces the cost. The low working temperature possess advantages such as lower power consumption, lower thermal induced signal shift as well as safe detection in certain environments where temperature is strictly limited.



## ACTIVE CENTRES FOR CARBON MONOXIDE CHEMISORPTION ON Pt-Sn/Al<sub>2</sub>O<sub>3</sub>

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Bimetallic Pt-Sn system have been widely used in the catalytic processes of reforming, hydrogenation, hydrogenolysis CO oxidation and as catalytic sensors. Usually the catalysts are prepared by impregnation of Al<sub>2</sub>O<sub>3</sub> with water solutions of H<sub>2</sub>PtCl<sub>6</sub> and SnCl<sub>4</sub>. Most often the first active component introduced is platinum followed by tin, however, for practical reasons it would be most convenient to introduce both components simultaneously. The difference in the preparation procedure may produce changes in the surface properties of the catalysts. One of the most often used methods for testing these properties is based on CO chemisorption.

The aim of this study was to characterise the interactions between CO and the active components introduced on the Al<sub>2</sub>O<sub>3</sub> support and check the effect of the catalysts preparation method on the CO chemisorption. For the sake of comparison also the catalysts Pt-Sn/Al<sub>2</sub>O<sub>3</sub> with tin introduced before platinum were tested. The paper reports the effect of the synthesis procedure of the catalysts PtSn/Al<sub>2</sub>O<sub>3</sub> on the type of surface platinum centres. The catalysts were obtained by the method of incipient wetness introducing either first tin followed by platinum after drying (first series) or platinum and then tin (second series) or two components simultaneously (third series). The catalysts performance was studied by CO sorption and the type and character of the chemisorption centres was determined by the FT-IR method.

The obtained results of our study have provided important information on the synthesis of the catalysts. The different sequence of the active phase introduction on alumina support leads to obtaining the catalysts with a certain amount of metallic platinum or platinum ions on the surface. The results indicated formation of different platinum centres: Pt<sup>0</sup>, Pt<sup>δ+</sup> (δ<sup>+</sup> ≤ 1), Pt<sup>m+</sup> (2 ≥ m > 1) and influenced on chemisorbed CO amount depending on the sequence of introduction of the active components.

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# LSMO MICROBOLOMETER FOR DETECTION OF THz RADIATION

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Astrophysicists claim that 98% of photons emitted since the Big Bang fall into THz range. So one can say that the space is immersed in THz energy. Nowadays the interest in this frequency region is coming not only from astronomy but also from the field of medicine (T-rays do not ionize atoms), gas analysis, security, etc.

Unfortunately, in the frequency range ~ 1-10 THz (the so-called THz gap) there is lack of detectors coming either from the side of “optics” or “electronics”. In such a case bolometers (that convert incident electromagnetic radiation into change in resistance caused by minute raising of temperature) seem to be a proper choice, because they are principally wideband detectors.

We decided to develop a microbolometer based on our two previous achievements: we found that the slope of temperature dependence of resistance in manganite perovskite in a certain temperature interval reached 11% per Kelvin [1]. The second achievement was the successful micromachining of GaAs for different kind of sensors (thermal converter).

The sensitive element is made of LaSrMnO<sub>3</sub> (LSMO) thin film deposited by dc magnetron sputtering [2]. The value of temperature coefficient of resistance (TCR) is about ten times higher than that of metals, which ensures sufficient sensitivity.

The bolometer consists of LSMO disk (diameter of 8 μm) situated at the feed point of a log-periodic antenna made of gold. The antenna is designed for the frequency range of 1.4 – 11 THz, i.e., it covers the above-mentioned THz gap.

The measuring stand and software for determination of basic bolometric parameters of the microbolometer prototype has been developed. The 3-ω method for determination of thermal conductivity of thin films has been implemented.

The bolometer is being developed under the grant APVT-51-032902 .

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## DEVELOPMENT OF A PARALLEL-COMPUTING EMBEDDED TELEMETRY SYSTEM FOR VOLTAMMETRIC MICROSENSOR AND BIOSENSOR APPLICATIONS

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A new parallel-computing embedded telemetry system for voltammetric microsensor and biosensor application is presented. The device, capable of driving two independent sensors, consists of a single-supply bipotentiostat- $I/V$  converter, a parallel microcontroller unit (pMCU), a signal transmitter, and a stabilized power supply. The system, derived from a previous design [1, 2], is built around a new pMCU (Parallax<sup>®</sup> Propeller<sup>™</sup>) consisting of eight 32-bit independent processors called COGs. The sensor conditioning waveforms are generated using a two-channel digital-to-analog converter (DAC) independently driven by two COGs. The resulting sensor current is converted to a digital value using two more COGs interfaced to a multi-channel analog-to-digital converter (ADC). The remaining COGs interface a keyboard, generate a video-composite signal (for monitoring sensor data using a local LCD screen) and drive a radio transceiver (for remote monitoring). The Propeller<sup>™</sup> firmware is developed in Spin, a specialized interpreted language, and Assembler then transferred to the pMCU through an in-circuit-serial-programmer (ICSP). The digital data is transmitted to a personal computer using a 2.4GHz XBee PRO<sup>™</sup>/IEEE 802.15.4 compliant transceiver with a line-of-sight range of up to 1000 m. A second XBee PRO<sup>™</sup> is connected to a PC via the Universal Serial Bus (USB). A completely customizable data acquisition software package (StampPlot<sup>™</sup> Pro) allows the PC to record, plot and display the received data. The design, construction and operation of the hardware and software are described. The system performance was evaluated *in vitro* using an automated dummy cell and a wide range of voltammetric microsensors (dopamine, ascorbic acid, nitric oxide) and platinum-based amperometric biosensors (glucose, lactate and glutamate). This device serves as a basic model to realize an *in vivo*, low-power, miniaturized telemetry system built with standard hardware components readily available. The introduction of parallel computing programming techniques could permit the development of low-cost devices, reduce complex multitasking firmware development, and offer the possibility of expanding the system simply and quickly.

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**POROUS NANOSTRUCTURED CERAMIC SENSORS OF HUMIDITY****I. UVAROVA***3, Krzhizhanovski str., 03142, Kyiv, UKRAINE**Institute for Problems of Materials Science of NAS of Ukraine, Kyiv, UKRAINE**e-mail: uvarova@materials.kiev.ua*

Everyday comfort, quality of food as well as technological processes of food industry and electronics, first of all, are determined by temperature and humidity. In order to measure humidity, sensors must meet the requirements for high sensibility and quick response in the wide humidity range, good repeatability and absence of hysteresis; suitable wear-resistance and resistance to soiling, slight dependence on temperature, and low cost and simplicity for preparation.

The influence of milling time and sintering temperature of the ceramic humidity sensors based on Al-Mg spinel and nanosized zirconium dioxide (specific surface area  $110 \text{ m}^2/\text{g}$ ) on the water adsorption and the change in the porous structure and resistivity has been studied. The optimal characteristics of Al-Mg spinels as ceramic humidity sensors were obtained after sintering the specimens at  $1200^\circ\text{C}$  for 94 h. In this case, resistivity against relative humidity is evaluated by a linear dependence; it changes by three orders of magnitude with increasing relative humidity from 20 to 80 %. Nanosized zirconium dioxide had the best characteristics after sintering at  $900^\circ\text{C}$ , when its resistivity changed more than three orders of magnitude with changing relative humidity from 20 to 80 %.

The specific surface area achieved a maximum after 96 h milling of aluminum and magnesium oxides mixture, and then it reduced. The kinetic data of water adsorption correspond to the changes of the specific surface area. The similar regularities kept after sintering of these powder mixtures.

The ceramic humidity sensors based on Al-Mg spinel and nanosized zirconium dioxide supplement each other since the resistivity of Al-Mg spinel changes against the relative humidity in the range of  $2 \cdot 10^9 - 3 \cdot 10^6 \Omega \cdot \text{cm}$  and the resistivity of zirconium dioxide changes from  $2 \cdot 10^7$  to  $1 \cdot 10^3 \Omega \cdot \text{cm}$ .

The simple method for preparation of Al-Mg spinel using only mixing and milling of the oxides is comparable with the traditional chemical methods for spinel production.

# ***BOOK OF ABSTRACTS***

## ***4. Posters***





# THE IDENTIFICATION OF THE PROPERTIES OF GRADED AND BIOLOGICALLY INSPIRED MATERIALS USING NANOINDENTATION

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Today the methods of detecting the properties of biologically inspired materials have to be developed as new materials are widely used in medical applications, for example in manufacturing artificial tissues for implants. As natural tissues have a complex structure (the most important characteristics of them can be analyzed only down to nanometer and micrometer scale), biomaterials require special methods of investigation.

The authors have been working on the problem of investigation of nanocoatings for a long time. In the work [1] the function of stiffness also known as Sneddon's function is used to identify the laws of changing of modulus of elasticity of a material. The formula for the Sneddon's function is

$$E_w = \frac{3}{4} \frac{P}{a\chi} \frac{1}{1-\nu^2},$$

where  $a$  is the contact area radius,  $\chi$  is the indenter displacement,  $\nu$  is the Poisson's ratio,  $P$  is the normal force. For a homogeneous material the stiffness is a constant equivalent to the material shear modulus [2] and for a nonhomogeneous one, the stiffness  $E_w$  depends on the size of the contact area  $a$

But in case when the thickness of a coating is unknown (as it usually arrives in biological structures), the stiffness curves may be superimposed on each other for different thicknesses and different laws of nonhomogeneity. In this case we need additional information, which can be provided by studying the surface outside the indenter. The present work describes the method of unambiguous definition of the material properties, starting from the form of the surface settling. This is an inverse problem to the one that was observed in [3].

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## **SMART MICROSYSTEM FOR DIAGNOSTIC IMAGING OF VULNERABLE PLAQUE**

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Smart Microsystems for Diagnostic Imaging in Medicine (SMiDA) is a project funded by the Research Council of Norway from 2004-2008. The objective is to develop robust technologies for medical diagnosis, in particular for identification of vulnerable plaque. A large percentage of heart attacks are caused by rupture of vulnerable plaques leading to the formation of blood clots, coronary stenosis and infarction. Technologies for reliable diagnosis of vulnerable plaque are not available today. In the SMiDA project methods are being investigated to identify chemical alterations in atherosclerotic tissue, to measure locally temperature, blood pressure and blood flow velocity, guided by 3D ultrasound imaging. The vision is to develop a smart sensor integrated in a catheter for intravascular imaging. Miniaturization is one of the main challenges of the project as the catheter can have a maximum diameter of 1mm. This tough requirement is being met by utilizing nanoscale CMOS technology. New technology is being developed to integrate the array of approximately 7500 capacitor micromachined ultrasound transducers (CMUTs) with the electronics.

To identify vulnerable plaque it is estimated that a depth resolution of 50-150 $\mu$ m into the artery wall is required. First iteration in the design of the CMUTs has resulted in the following parameters: 5.7 $\mu$ m radius, 100nm membrane thickness, vibration amplitude in the nanometer to picometer range and approximately 35MHz center frequency. New processing techniques have been developed to fabricate these minute structures. The CMUTs are fabricated by transfer of the silicon nitride membrane with a silicon dioxide layer to a silicon wafer with pre-etched cavities using hydrophilic fusion bonding.

The SMiDA project is an exciting multidisciplinary project requiring research in the field of medicine, physics, electronics and optics. Support from the electronic and ultrasound industry as well as close contact with cardiologists has been of great value.

## **MEASURING THE PROTEIN BINDING CAPABILITY OF BIOCOMPATIBLE SURFACES WITH LABEL-FREE BIOSENSOR**

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The aim of the present work is to measure the adherence properties of serum albumin and fibrinogen on various surfaces (stainless steel and its modified variations) with sampling and real-time measurement.

An OWLS120 instrument (MicroVacuum, Hungary) was used to determine the binding of proteins to different surfaces. Optical waveguide light mode spectroscopy is a label free biosensor method based on the resonance incoupling of a polarized light with a grating into a planar waveguide. It can characterize in real time the adsorption/desorption of molecules that affects the refractive index of the sensor. (Ref. 1)

Experiments were carried out in a continuous flow system where a syringe pump ensured the flow and the analytes were passed in through an injector valve. Various cuvettes were proved to study how the geometric configuration of the flow cell effects the measurements.

The amino functionalized sensor surface was activated with glutaraldehyde and the protein (BSA, fibrinogen) or the antibody was coupled covalently depending on the measurement method.

The point of sampling is to dip the sample into the buffer and to measure the concentration of the supernatant from time to time. In the course of the real-time measurement the change of the concentration can be measured constantly.

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**PREPARATION OF ENFET TYPE GLUCOSE BIOSENSOR**

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Enzymes conjugated with gold nanoparticles can be efficiently used in biosensor applications. These nanoparticles enhance the amount of the adsorbed enzymes due to their large surface area and surface free energy [1]. Moreover, gold nanoparticles provide a biocompatible environment so that enzyme activity can be retained at the end of the immobilization process. Gold nanoparticles can form strong covalent bonds with  $-NH_2$ ,  $-SH$  and  $-CN$  functional groups. This property enables to form multilayer films composed of enzymes, gold nanoparticles and cross-linker carrying above mentioned functional groups. In addition, these nanoparticles make the charge transfer between the enzyme and electrode simpler [2].

Sol-gel methodology can be used to produce three-dimensional matrices for immobilization of enzymes. These gels provide adjustable porosity, chemical inertness and negligible swelling in aqueous environment [3].

In this study, indium oxide coated glass substrates have been covered with silica layers. Into the pores of this sol-gel network, gold nanoparticles have been adsorbed. Then by layer-by-layer covalent attachment of gold nanoparticles with enzymes via a cross-linker (cycteamine), an ENFET biosensor for the detection of glucose will be developed.

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**CARBON NANOTUBES AS BIOSENSORS**  
**STABILITY OF CARBON NANOTUBES IN HUMAN BLOOD PLASMA**

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The creation of biosensors on the base of iron carbon nanotubes is one of the advanced scientific directions of biomedical nanotechnologies. The first steps in diagnostics and treatment of cancer with using carbon nanotubes demonstrated their advantage over today methods of X-ray analysis, puncture and others.

In this case, the interaction of nanotubes with antibodies, which are used in biomedicine for diagnostic of cancer decease, has been studied. Immune system generates antibodies, as response to the appearance of foreign antigens in organism. Taking into account advantages of carbon nanotubes as antibody carriers it is necessary to investigate their corrosion stability in biological media, especially in human blood plasma.

Interaction of nanotubes prepared by reduction of carbon monoxide with human blood plasma and physiological solutions (0,9 % NaCl, Ringer, Ringer - Lokk) has been studied. It was shown that carbon nanotubes containing 33 and 50 mass % of iron are stable enough both in human blood plasma and in physiological solutions. The iron concentration in human blood plasma changed slightly after the interaction.

Magnetic characteristics of carbon nanotubes and sorbtion capability towards methylene-blue do not practically change after interaction with human blood plasma.

Thus, iron carbon nanotubes with 30-50 mass % iron slightly interact with human blood plasma and can be used as advanced carriers of specific antibodies for identification of cancer center in living organism.

## **MONITORING ENVIRONMENTAL IMPACTS OF SURFACE MINING USING SPOT HIGH RESOLUTION VISIBLE (HRV) SENSOR SYSTEMS**

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In surface coal mining large volumes of waste material are excavated and removed from one place to another causing continuous change in topography with time. This requires systematic topographical measurements and monitoring of the mine environment to update the pit plans. Also, the impacts of mining activities on environment is deteriorious and of vital concern for sustainable development. Therefore, it is essential to monitor the environmental effects of mining periodically and to detect changes due to mining in the region in terms of topograpgy, vegetation, and drainage pattern. Conventional methods, such as surveying, do not provide robust and rigorous algorithms to serve the mine monitoring objective.

The present study explores the possibility of using high resolution visible sensor in monitoring the changes in mining area in terms of estimating the areal extent of overburden stripped in a surface lignite mine and the environmental impacts of the surface mining activities. For this purpose two satellite images (SPOT Panchromatic and Multispectral Images) covering Göynük Lignite mine area acquired in the years 1987 and 1999, were used. The areal extent of excavated overburden was calculated by means of overlay analysis using Digital Elevation Model (DEM) of the area and available topographical surveys.

This study also presents the land cover mapping of the mine area in accordance with the change detection techniques. After preprocessing; i.e. radiometric and geometric correction of the images, change detection is accomplished by comparing two images by means of band differencing algorithm. The change in the region, which occured between the years 1987 and 1999 due to the mining activities, is detected as an areal extent. The primary interests in the change detection study are to identify the areas affected from mining activities and the direction of progress of mining. The results of the band differencing algorithm show that most of the changes occurred in the western part of the mine region between the years 1987 and 1999. Also, on the basis of supervised classification technique, using the multispectral SPOT image acquired in 1999, the mining region is analysed quantitavely with regard to the residential area, vegetational area, bare soil and mining area.

The accuracy assessment of the classification is performed by using the field observations done with Global Positioning System (GPS). Increasing the spatial resolution in addition to the spectral resolution by merging the panchromatic and multispectral images increased the classification accuracy. 88% of accuracy obtained for the merged image in land cover mapping indicated that there is a high potential in using merged panchromatic and multispectral SPOT images for this purpose.

## PHTHALOCYANINE COATED QCM SENSORS FOR MONITORING POLLUTANTS IN AQUEOUS MEDIA

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Regulations on the maximum concentrations of many environmental pollutants in air and water are imposed by governments worldwide to minimize risks to humans, animals, and the environment creating the need for monitoring and control. Currently applied monitoring and analysis methods are, however, often costly and can only be used off-site. Thus, much of the costs connected to environmental pollution are caused by laboratory analysis. Additionally, such off-site analysis can be negatively affected by transport and storage of the samples. Consequently, there is a high potential for technologies allowing inexpensive long-term monitoring and accurate analysis of environmental contaminants [1].

Chemical sensors are prospective solutions meeting those goals. Especially, quartz crystal microbalance (QCM) based sensor systems have been suggested for this application field, as they are simple to use, inexpensive, fast, and suitable for in-field measurements [2]. Moreover, phthalocyanines (Pc) have proven their excellent sensing capabilities already in many gas sensing applications with QCMs and other types of sensors [3]. In this work water insoluble Ni phthalocyanines (NiPc) are employed first time to aqueous media in which they can be equally well used. A commercial QCM measurement device (KSV Instruments QCM – Z500) equipped with 5 MHz QCM sensors was used for the tests. The sensors can be driven at their fundamental frequency or overtone frequencies. Coatings were deposited using the jet-spray method. As test analytes 1,2,4-trichlorobenzene, chloroform and toluene were selected.

The new NiPc based layers proved in direct comparison to be more advantageous for later use over layers already known as being sensitive to the analytes listed above, e.g. different calixarenes, especially in terms of sensitivity, response and recovery times. From the measurements a detection limit near 2 ppm was extracted for toluene and chloroform and at sub-ppm level for 1,2,4-trichlorobenzene. Full sensors response is reached in 20s (stop-flow). The sensor response and baseline are stable over long time making repeated measurements possible over a time frame of several weeks. Concluded from all available data, the NiPc used the first time in liquid media show a promising performance for future use in sensor systems.

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# INFLUENCE OF ADSORPTION OF ACTIVE MOLECULES ON ELECTRICAL TRANSPORT IN NANOSTRUCTURED POROUS SILICON

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Porous silicon (PS) prepared by electrochemical etching of crystalline silicon is a model object to study the adsorption induced electronic phenomena in ensembles of semiconductor nanocrystals [1]. Because of a huge specific surface area (up to  $10^3 \text{ m}^2/\text{g}$ ) the electronic and optical properties of PS are extremely sensitive to the environment of Si nanocrystals in PS. The strong effect of molecule adsorption on the physical properties of PS allows us to use PS for applications in gas sensors [2]. In our work the adsorption effect of active (donor and acceptor) molecules on the charge carrier transport in heavily doped PS layers is investigated.

PS films were formed from (100)-oriented p-Si:B wafers ( $\rho=1\div 5 \text{ m}\Omega\cdot\text{cm}$ ) and n-Si:As ( $\rho=1\div 5 \text{ m}\Omega\cdot\text{cm}$ ) wafers by electrochemical anodization in a solution of HF (48%) in ethanol (1:1) at a current density of  $30 \text{ mA}/\text{cm}^2$ . Free-standing  $50\text{-}\mu\text{m}$ -thick PS films were obtained by lifting during a short electropolishing step at a current density of  $500 \text{ mA}/\text{cm}^2$ . For electrical measurements the samples were provided with evaporated Al contacts. The dc and ac conductivities were measured using a Keithley 6487 unit and an impedance-analyzer HP 4192A. Measurements of the IR absorption spectra of PS were carrying with a Perkin Elmer RX I spectrometer. The free carrier (holes or electrons) concentrations in PS are determined by analyzing of the IR absorption spectra according to Ref. [3]. The motilities of electrons ( $\mu_e$ ) and holes ( $\mu_h$ ) are calculated by using data on the conductivity and free carrier concentration [3]. The adsorption of donor ( $\text{NH}_3$ ) and acceptor ( $\text{I}_2$ ) molecules on PS have been done at room temperature. Our experiments showed that the molecule adsorption results in strong changes of both the electrical conductivity and mobility. The samples in vacuum are characterized by  $\mu_e \approx 1.1 \cdot 10^{-2} \text{ cm}^2/\text{V}\cdot\text{s}$  and  $\mu_h \approx 2.9 \cdot 10^{-3} \text{ cm}^2/\text{V}\cdot\text{s}$  for of n-type and p-type PS, respectively. The charge carrier mobility and conductivity were found to increase by several orders of magnitudes after of the corresponding molecule adsorption (donor or acceptor molecules for n-type or p-type, respectively). These results demonstrate possibilities to employ heavily doped PS for sensor applications.

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## **NEW ROLES FOR IFT IN CELL FUNCTION**

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Discoveries on the mechanism of assembly of cilia (flagella) in the lowly, biflagellated, eucaryotic green alga *Chlamydomonas* have triggered a renaissance of interest in the organelles along with the recognition of their key sensory roles in nonsensory tissues. *Chlamydomonas* researchers uncovered an entirely new set of cellular machinery essential for transporting the protein components of cilia and flagella in all ciliated/flagellated eukaryotic cells between their site of synthesis in the cell body and their site of assembly at the tip of the flagellum (intraflagellar transport: IFT) [1].

IFT was first observed in *Chlamydomonas*, and orthologs of some of the polypeptides involved in IFT have recently been identified in other organisms, including *C. elegans* and the mouse. In addition to a role in the assembly and maintenance of cilia and flagella, evidence is reviewed here that indicates defects in the process of IFT may be related to problems with human health. Moreover, recent data suggest the possibility of two new roles for IFT in cell function. The first is in transcriptional control of the genes encoding ciliary and flagellar proteins. IFT could provide a mechanism whereby the cell senses the presence or absence of its cilia or flagella and responds by turning on gene transcription resulting in replacement of the missing organelle. The second role is in signal transduction, whereby cilia act as sensors of the external cellular environment and transduce information about the surroundings into intracellular signals that are sent via IFT to the cell body, thus inducing an appropriate cellular response to the environment [2].

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## **GRAVITY-INDUCED CHANGES IN CORTICAL MICROTUBULES IN ARABIDOPSIS THALIANA ROOT CELLS**

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Biological systems depend on gravity by way of the bifurcation properties of certain types of non-linear chemical reactions which are far-from-equilibrium. MTs are long cylindrical polymers that exhibit polymerization through the addition and release of  $\alpha\beta$ -tubulin dimers at their ends. This “treadmilling” drives microtubule repositioning and the reorganization of microtubule arrays. Microtubules are chemically anisotropic, growing and shrinking along the direction of long axis. This leads to the formation of chemical trails, comprised of regions of high and low local tubulin concentration from their shrinking and growing ends respectively. Gravity acts by way of its directional interaction with the macroscopic density fluctuations present in the solution arising from microtubule disassembly at the instability. Space experiments show gravity dependence of microtubule self-organization *in vitro*.

We decided to check the effect of the gravitational field on the MT self-organization *in vivo* experiments. To modulate some biological effect of microgravity on plant cytoskeleton we used clinorotation (2rpm). Microtubules were visualized by using a stably transformed line of transgenic *Arabidopsis thaliana* expressing GFP-MAP4 fusion protein. We report changes in the microtubule spatial configuration under clinorotation in epidermal and cortical cells in distal elongation zone of roots, where numbers of discordant shorter microtubules appeared. To reveal a dynamics of this realignment we applied 5 $\mu$ M/L oryzalin (an antimicrotubule drug that binds to tubulin dimers and blocks following polymerization) in control and clinorotated seedlings. Combined application of clinorotation and oryzalin did not result in additive effects and evoked disruption of tubulin cytoskeleton. Rapid oryzalin treatment, followed by clinorotation, revealed that disoriented microtubules, which appeared in distal elongation zone under clinorotation, are hypersensitive to oryzalin compared to other microtubule populations in root.

As far as oryzalin blocks the addition of tubulin dimers to the growing end of a microtubule, a dynamic microtubule with high turnover will be more sensitive to such drugs as compared to a stable microtubule with a low rate of dimer exchange. The increased oryzalin sensitivity of these microtubules can be interpreted as consequence of the increasing in rate of microtubule dynamic turnover. We suppose that dynamics of these microtubules depends on the activity of structural microtubule associated proteins, specific for distal elongation zone. Cells of this zone accomplish a developmental transition recently from cytoplasmic driven expansion to vacuole driven elongation, which results in switch from isotropic to anisotropic pattern of growth. Our results suggest that cortical microtubule arrays in cells of the distal elongation zone are influenced by altered gravity due to special physiological properties of cells in this zone, probably, because of a highly dynamic cortical interphase microtubule arrays. It is assumed that the changes in the stability of the tubulin cytoskeleton may underlie the gravity-induced rearrangement of cortical microtubule arrays. It means that self-organization of cortical microtubules may be considered as gravity sensor in cells, which are not specialized to perceive gravity.

**DIRECT INVESTIGATION OF BACTERIOPHAGES-BACTERIA  
INTERACTION**

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Number of publications devoted to atomic force microscopy (AFM) study of biological objects is growing presently owing to its unique advantages over conventional microscopic techniques. The most remarkable feature of AFM, high resolution imaging of soft living objects, allows studying various pathogens – bacteria, viruses, and their interactions with host surfaces without special pre-deposition preparation. In this report we present new protocols that have elaborated for visualization of interaction of bacteriophages with bacterial cells. This protocol allows monitor the process of cell - bacteriophage interaction on time scale from several minutes to several hours. We illustrate our results on the example of interaction *E.coli* and *S. typhimurium* cells with bacteriophages.

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**NANOCOMPOSITS FOR ACTIVE ELEMENTS  
OF HUMIDITY'S SENSORS****Lyubov KUNYTSKA**

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Inorganic and organic- inorganic protonconducting materials are received by of a sol-gel method of synthesis. Conductivity in them is realized due to protons which are formed of mobile hydrogen of phosphoric acid groups which contains in structure of hybrid organic-inorganic nanocomposites and mobile protons of acid groups which contain in its organic component. Such materials show high levels of conductivity (up to  $10^{-2}$  Sm/cm) in the temperatures range of 293 – 353 K and are perspective for use as materials for electrically active elements of sensor controls which functionate by the phenomenon of ionic (proton) conductivity. Laboratory samples of sensor controls of humidity which impedance characteristics have been investigated in a range of frequencies from 10 up to  $10^5$  Hz in conditions of controllable temperature and humidity. It was founded that the variation of relative humidity from 20 up to 60% at 293 K results in reversible changes of ionic conductivity from 10 up to 40% depending on nanocomposite membranes structure. Depending on frequency of measurement value of complex conductivity changes from 400 to 2000 in a studied range of relative humidity conditions. The optimal appeared a range of frequencies is 0.1 – 1 kHz.

*Key world:* sol – gel nanocomposits, humidity sensors, fractal dimension, ion conductivity

## **CARBON COMPOSITE ELECTRODES APPLIED FOR ELECTROCHEMICAL SENSORS**

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Electrodes based on particulate carbon-epoxy or polystyrene composites, have been formed and characterized using electrochemical methods and scanning electron microscopy. The composites materials are produced by film casting from a toluene solution. The film was pressed at 250 °C in a hot press (Fontaijne, The Netherlands) and cooled to room temperature in the open air. Working electrodes made of various forms of graphite or carbon and using different polymers, by type of exfoliated graphite-polystyrene, exfoliated graphite-epoxy, expanded and exfoliated graphite-epoxy and carbon nanofiber-exfoliated graphite-epoxy were tested for electrochemical sensing of various organic pollutants in aqueous solution.

The prepared carbon composite electrodes showed good mechanical strength, low electrical resistivity, and easy renewable surface by simple polishing, useful characteristics for electroanalytical purposes.

The electrochemical performances of these electrodes were studied by cyclic voltammetry (CV), linear-scan voltammetry (LSV), chronoamperometry (CA) and differential pulsed voltammetry (DPV). All measurements were carried out using a potentiostat/galvanostat PGSTAT 302 (Eco Chemie, The Netherlands) controlled with GPES 4.9 software and a three-electrode cell (Metrohm, Switzerland), with a saturated calomel electrode as reference electrode, a platinum counter electrode and carbon composite working electrode. The voltammetric responses of these materials are similarly of that found for a microelectrode array, in which "edge effects" contribute significantly to the Faradic current. However, these arrays are randomized, in that the size, shape and inter-electrode separation are distributed over a wide dimension range, in accordance with the structural aspects observed by using Scanning Electron Microscope (Philips CM30T).

Some mechanistic details concerning the oxidation of the tested organic pollutants on the used carbon composite electrodes were evaluated. Calibration plots showing a linear relationship between the amperometric response of the carbon composite electrode and pollutant concentration were obtained in various concentration ranges. The analytical parameters relating the amperometric detection of these pollutants, *e.g.*, the electrode sensitivity, the correlation coefficient, the lowest limit of detection depended on the type of the carbon composite electrode and the used technique.

Electrodes manufactured using a mixture of particulate carbon and a resin represent an attractive approach to the fabrication of various amperometric sensors, whose surface can be easy renewed and reproduced by simple polishing.

## **InO<sub>x</sub> LAYERED SURFACE ACOUSTIC WAVE (SAW) DEVICES FOR GAS SENSING APPLICATIONS**

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Layered metal oxide surface acoustic wave (SAW) gas sensors have recently received increased attention for gas sensing applications. They have higher sensitivity comparing with their non-layered SAW counterparts due to the fact that the interaction of reducing and oxidizing gases with the metal oxide selective layer perturbs the electrical boundary condition at the surface of the SAW device. As a result the propagation characteristics such as the velocity and amplitude of the electromechanical waves are affected.

In this work DC sputtered InO<sub>x</sub> thin film, approximately 100nm thick, was deposited as selective layer onto commercially available SAW filters operating in a wide range of central frequencies ranging from 139MHz-930MHz. The filters responses were tested over a range of operating temperatures, and towards oxidizing and reducing gases such as NO<sub>2</sub> and H<sub>2</sub> respectively, with varying concentrations. Strong frequency shifts as high as 300KHz and 600KHz were observed towards 600ppm H<sub>2</sub> and 8.5ppm NO<sub>2</sub> respectively, at 160°C operating temperature, for the 930MHz SAW filter.

In addition, a thorough examination of InO<sub>x</sub> thin films was carried out towards different O<sub>3</sub> concentrations ranging from 15ppb – 3ppm in order to study the lower sensitivity limits of InO<sub>x</sub> towards O<sub>3</sub>. The films were then deposited onto XY LiNbO<sub>3</sub>/ZnO Saw sensors and exhibited high sensitivities with fast responses. In particular a frequency shift of 31.5KHz for 100ppb O<sub>3</sub> and 78.5KHz for 25ppb O<sub>3</sub> were observed for the 100nm sensor.

## TECHNOLOGY OF OBTAINING CARBON NANOTUBES

**G. OSPANOVA, M. TLEBAYEV, E. KOLTSOVA, T. BAYZHUMANOV**

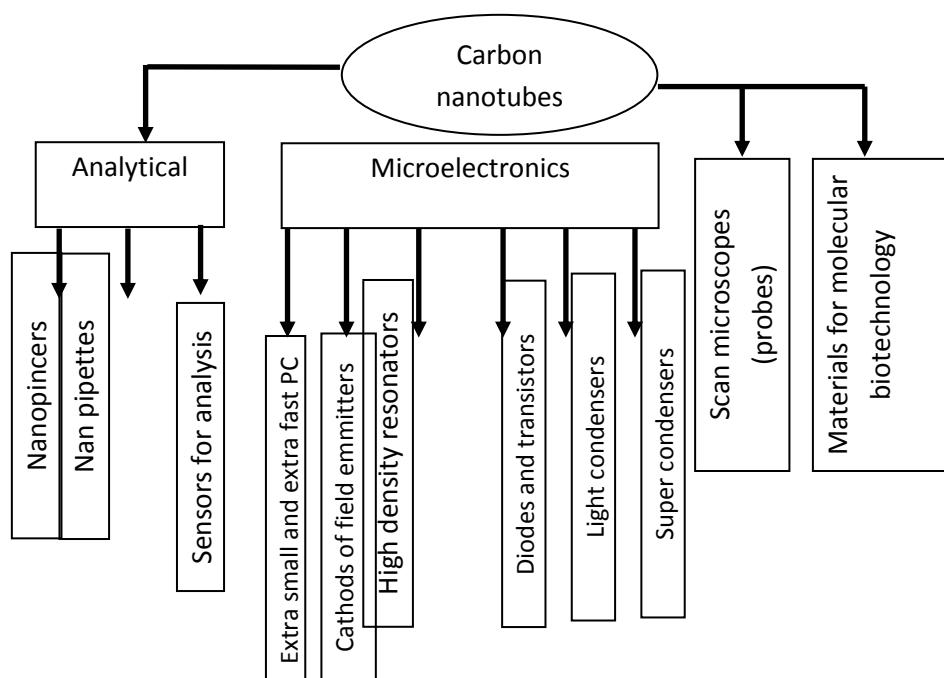
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Now carbon nanotubes are the advanced materials. Methods of hydrocarbon catalytic pyrolysis are very perspective for obtaining of carbon nanotubes and nanofibres.

Methane catalytical pyrolysis technology with obtaining of carbon nanotubes and nanofibres is developed. Kinetic scheme of carbon nanotubes and nanofibres formation from methane is proposed. Mathematical model of methane catalytic pyrolysis is developed. Equipment for methane catalytical pyrolysis is created.

Economic effectiveness of methane catalytical decomposition with obtaining of carbon nanotubes, nanofibres and hydrogen is calculated. It is shown that carbon nanotubes cost for 1 kg is \$11-18; hydrogen cost depends on its purity is \$ 22-82.

Carbon nanotubes usage are huge. Application of carbon nanotubes is shown below.





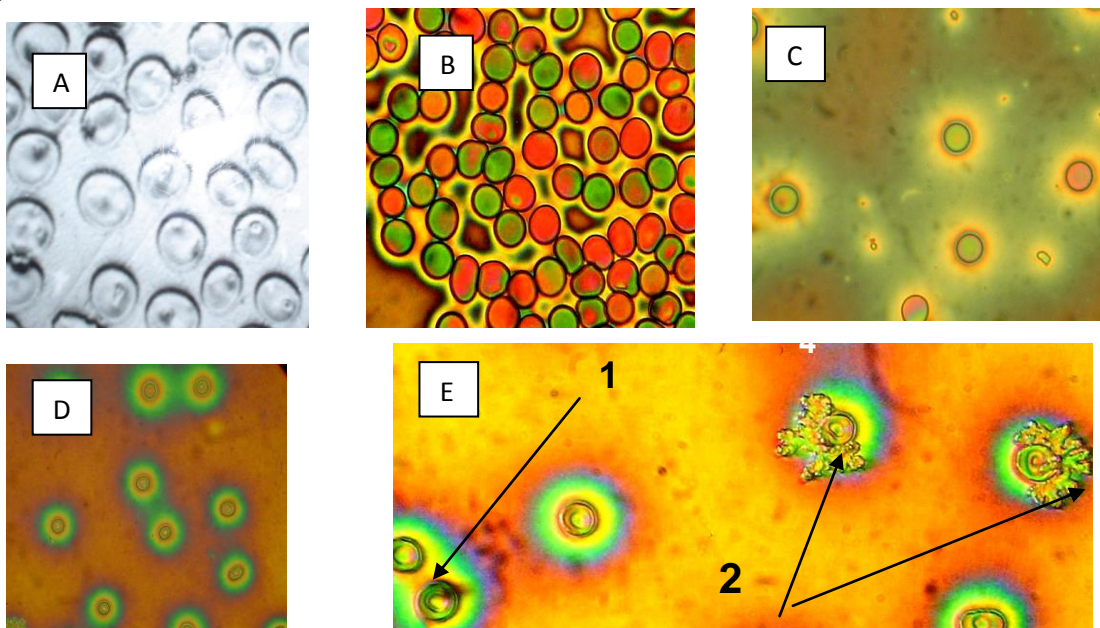
# **VISUALIZATION AND DETECTING OF CHEMICAL COMPOUNDS IN BLOOD SMEARS**

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Usually to determine chemical compounds of sample and its quantity need to use very complicate and expensive spectroscopic methods. In present paper we offer the idea of the new approach not only detecting some chemical compounds as well as its visualization to see in color a difference areas of the sample. For instance to get color image of morphological structure of blood smears are usually using staining of living blood smears by dyes. But this method leads to disruption. Usually in medical practice conventional bright-field microscopy let us see black and white image of separate morphological elements of blood smears only (Figure 1A). Proposed the new nondestructive method of optical microscopy capable of examining the structures of living cells in their natural colors without staining them, using a specially designed substrate for deposition of biological sample and observing native structure in reflected light. This method based on physical phenomena of white light interference reflected from sample surface and special supporter on which this sample is deposited. It allows to occur at the image plane converting previously invisible gradients of refractive index within the specimen in to intensity gradients in the



the image. Color interference contrast image is achieved due to special condition. If any conditions of the experiment are fixed beside of chemical content of sample we can say that color contrast is caused by chemical compounds. On fig.1(B) red color of erythrocytes correspond hemoglobin. Therefore from this picture we can see visually content of hemoglobin in blood smear. Similar we can determine other chemical compounds after calibration color scale by alternative methods. To demonstrate the potential usefulness of this method, we provide qualitative data describing color image of healthy and pathological damaged cells for alive and dry blood smears (Figure 1A-1E).

## **IN VIVO OBSERVATION THE SUPER WEAK LUMINESCENCE OF SINGLE LIVING COTTON CELL**

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In first super weak luminescence of separate living cotton hair-cell was investigated for cotton plant variety *Tashkent - 1*, *C- 4727*, *Gossipium hirsutum*, *Turfan guza* *G.herbaceum*, *C-6063* *G. barbadense* *L.* and wild form *G. raimondii*. This phenomena under optical microscope Neophot-2 was observed with using so named replica–reprint technique for preparation living cotton cell. For this purpose the polymer solution (gelatin) was used for visualization of track luminescence in bulk of deposited polymer film on cotton cell-hair [1]. It is showed that luminescence for lifeless and mature plant cells were not generated but maximal luminescence is detected from apical parts of cotton hairs in early growth stage (4-6 days after flowering). By us it is showed that luminescence have cone like form difference variety of cotton plant (Figure 1). This fact indicates to a focusing of radiation connected with morphological and structural features of apical part of cotton cell-hair at early stages of their evolution. The electromagnetic nature of cotton cell luminescence has been showed by experiments with using of the photoelectric multiplexer (PEM) sensing to an ultraviolet radiation. At insertion in darkness of cotton seed-bud on a window PEM the dark current increased on 6-8 %. Actually radiation flux from developing cotton hair is more high-power, as through a window of PEM the small part of radiation was fixed only. Therefore on the basis of a large experimental material obtained by many investigators on miscellaneous biological objects it is possible to talk that the luminescence of cotton hairs are high energy ultra-violet irradiation.



Figure 1. Spontaneous light luminescence from apex of separate cotton cell-hairs (5-7 days after flowering)

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# USE OF NMR SPECTROSCOPY AND ZEOLITE CRYSTALLITE MATERIALS TO STUDY OF BENZENE ADSORPTION KINETICS

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At this work we present a results of experimental study of benzene adsorption kinetics with use of NMR spectroscopy and zeolite crystallite materials with the subsequent mathematical modelling of such process and numerical computer simulation based on the experimental data.

The idea of experiment part is to select successively several sections at different positions of the sample along the Z axis. In this way one obtains for each position a spectrum corresponding to the composition of the sample at this point, which proves to be very interesting in the case of heterogeneous samples, and especially for the diffusion of gas before the equilibrium of adsorption. In order to perform these experiments a device was designed to displace the sample in the magnet by increments of the order of the micrometer at the lowest. The mathematical model of benzene diffusion kinetics in the zeolite catalytical bed (which we consider to be an heterogeneous and multilayers porous media) is defined by the solutions of the system of differential equations:

$$\varepsilon_{inter_k} \frac{\partial c_k}{\partial t} = D_{inter_k} \cdot \varepsilon_{inter_k} \frac{\partial^2 c_k}{\partial z^2} - \frac{3(1 - \varepsilon_{inter_k})}{R} \cdot (D_{int ra_k} \frac{\partial q_k}{\partial r})|_{r=R}; \quad (1)$$

$$\frac{\partial q_k}{\partial t} = D_{int ra_k} \left( \frac{\partial^2 q_k}{\partial r^2} + \frac{2}{r} \frac{\partial q_k}{\partial r} \right) \quad (2)$$

corresponding to the mass balance in the macropores ( $c_k$ ) and in the micropores ( $q_k$ ) for each  $k$ - layer .

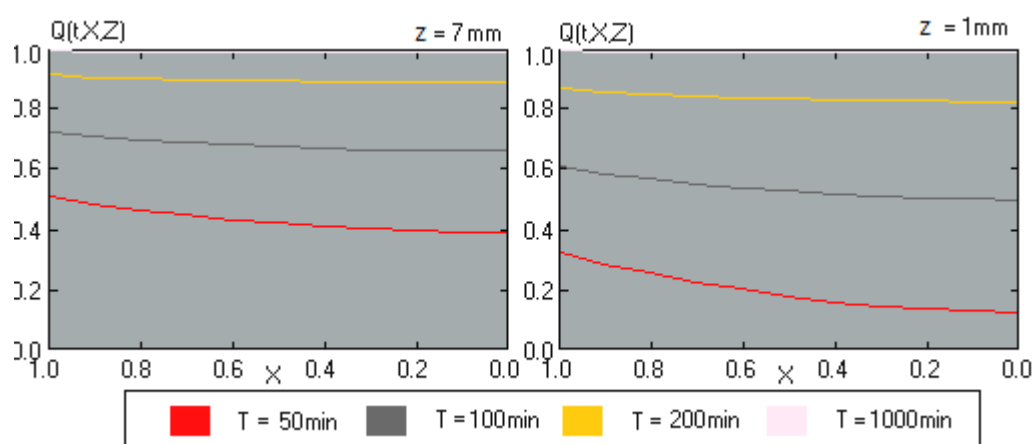


Figure 1 -  $C_6H_6$  - concentration profiles in micropores  $Q(t, X, z)$ , in zeolite crystallites localized at different positions  $z$  (mm) in the bed, and for different times  $t$  (min)

## THE ANALYSIS OF CATALYTIC ACTIVITY OF Pd-SnO<sub>2</sub> GAS SENSING MATERIAL IN OXIDATION REACTION

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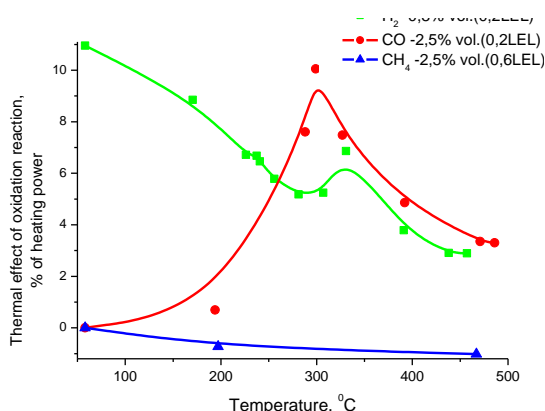
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The target of present work was the study of (1) the interference between catalytic oxidation of reducing gases on the surface of semiconductor gas sensor and (2) the correlation between the oxidation processes and conductivity response of the sensor. The first process can lead to the overheating of the sensor and to its damage, whereas the correlation between oxidation rate and semiconductor response can enable us to reveal the mechanism of sensor response on Pd doped tin dioxide.

In this work, we used thick film gas sensing layer based on tin dioxide (~55 m<sup>2</sup>/g) superficially doped with palladium (3 wt. %). Thick film sensing layer was deposited on Al<sub>2</sub>O<sub>3</sub> substrate, on the backside of the substrate Pt-based heater was formed by screen printing technology. Relatively low power consumption (~ 220 mW at 450°C) makes possible the thermochemical measurements with the sensor. The sensor response was determined for H<sub>2</sub>, CO, and CH<sub>4</sub> gases at the concentration equal to 0,2-0,6 Low Explosion Level (LEL). Thermal effect and, therefore, the oxidation reaction rate on the surface of the catalyst were determined at constant temperature of the sensor. For this, a decrease in heating power, necessary to maintain constant temperature, was measured.

The measurements were carried out in a temperature range from 50 to 500°C. The thermal effect of the reaction on the catalyst as a function of temperature is shown in Fig. 1.



The results show that the position of maximum of thermal effect of the reaction corresponds, in general, to the maximum of the sensing layer response (sensor response is expressed as a ratio  $(G_g - G_0)/G_0$ , where  $G_g$  and  $G_0$  are sensing layer conductivity in gas and in air, respectively) measured earlier under isothermic conditions.

*Fig. 1. Thermal effect of the H<sub>2</sub>, CO, and CH<sub>4</sub> oxidation as a function of temperature. Gas concentration is shown in the plot.*

Local maximum for H<sub>2</sub> curve and maximum of thermal effect for CO curve correlates with the interval of co-existence of two palladium oxides: PdO and PdO<sub>2</sub>, found earlier in our experiments. The results obtained in this work show clear difference in the catalyst activity in the reactions of CO and H<sub>2</sub> oxidation in low-temperature range. This difference can be used for selection between CO and H<sub>2</sub> by semiconductor gas sensor operating in pulse heating mode. In this mode, the sensing material is excited thermally at 450°C, and the measurements are performed at about 100°C.

## **ELECTROANALYSIS OF REDOX-INACTIVE PROTEINS BY LIQUID FILM-MODIFIED ELECTRODES**

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The electrochemistry of interface between two immiscible liquids became an important tool of modern analytical chemistry. Accordingly, the transfer of redox-inactive ions through the interface gives the amperometric response similar to the Faraday processes at metal-solution interface.

To observe protein transfer across liquid|liquid interface it is necessary to form water-in-oil microemulsion with solubilized protein stabilized with surfactant molecules. Generally micelles, which are able to contain proteins, can be formed exclusively in non-polar organic solvents. Therefore we developed an improved electrochemical setup based on solid electrode shielded with thin layer of redox polymer solution in organic solvent [1]. Novel system allows operating with non-polar organic solvents and as a result observing electrochemistry of protein transfer through liquid|liquid interface.

Dramatic increase of current which exceed two orders of magnitude with respect to pure buffer has been obtained by cyclic voltammetry after incubation of electrode in buffer solution of redox-inactive protein. Background voltammograms recorded in the absence of surfactant in organic phase did not display any raise in redox-activity indicating no solubilization of protein by redox polymer. An increase of current has not been observed with polar organic solvent dichloroethane, which is due to the control of droplet dimensions of water-in-oil microemulsion by the dielectric permittivity of organic solvent. The additional proof of protein extraction into thin organic layer has been obtained by the kinetic measurements of enzymatic activity in organic phase.

Varying the protein nature and experimental conditions some understanding of electrode process has been achieved. The voltammetric peak current was proportional to the protein concentration from 5  $\mu\text{M}$  to 200  $\mu\text{M}$  for the most of proteins, which is attractive for analytical applications [2]. The opposite charge of protein globule and surfactant molecules led to stronger current increase, which illustrates an important role of protein-surfactant interactions in response emergence. Employing electrochemical impedance spectroscopy, better sensitivity and lower detection limits to proteins have been obtained

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## **OPTICAL SPECTROSCOPY OF SILICON NANOCRYSTALS FOR BIOMEDICAL APPLICATIONS**

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Electronic and optical properties of silicon nanocrystal (nc-Si) assemblies prepared by electrochemical methods differ drastically from those of the bulk Si (c-Si). Besides the quantum confinement the properties of nc-Si of 1-5 nm sizes are strongly affected by surface treatment and molecular ambient. There are hopeful prospects for using silicon nanocrystals in photodynamic cancer therapy. For this biomedical application one needs nc-Si-based materials with controlled bioactive properties. Our work demonstrates the required approach to form such materials, which possess desirable structural, electronic, optical and bioactive properties.

The approach employed in present work is based on the electrochemical porosifying of c-Si wafers of certain specific resistivity in hydrofluoric acid solutions. The prepared free-standing porous Si (PSi) films were dried in air and then milled to obtain nc-Si powders. The nc-Si powder was dispersed in pure water bubbled with oxygen to form homogeneous aqueous suspensions. The dry PSi powder was investigated in vacuum and in ambient of oxygen molecules. The photoluminescence (PL) properties of nc-Si have been investigated and explained by the radiative recombination of excitons confined in the nanocrystals. The excitons are found to transfer non-radiatively their energy to oxygen molecules adsorbed on the surface of nc-Si. The PL spectroscopic experiments clarify that the energy transfer is mediated by the direct resonant electron exchange between photoexcited Si nanocrystals and oxygen molecules adsorbed on their surfaces. The singlet oxygen (SO) generation is observed for nc-Si both in gaseous and liquid ambiances. In particular, the PL intensity of nc-Si in oxygen-saturated water decreases by factor of 1.5-2 in comparison with that in oxygen-free water (or in vacuum). This fact indicates a high quantum efficiency of the SO generation. Our results have shown that the photosensitization of SO by nc-Si dispersed in water can be used in photodynamic cancer therapy.

**SYNTHESIS OF PURE AND DOPED WO<sub>3</sub> NANOPARTICLES BY  
FLAME SPRAY PYROLYSIS AND THEIR GAS SENSING  
PROPERTIES**

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As one type of wide-bandgap semiconducting metal oxides, tungsten oxide has attracted a lot of attention due to its promising property in gas detection behavior, esp. in NO<sub>x</sub> and NH<sub>3</sub> detection. To improve the sensing performance of a material, acquisition of larger surface area and doping of other metal elements are two feasible routes.

In this paper, by using flame spray pyrolysis method, large scale of monoclinic structured tungsten oxide nanoparticles, with an average diameter of 30 nm, were successfully synthesized. A series of dopants, including Co, Cr, Ce and Mn, were also added respectively by changing the precursors. All products were heat treated at 500 °C afterwards. The as-prepared and heat-treated products were characterized using transmission electronic microscopy, X-ray diffraction, nitrogen adsorption and Raman spectroscopy to determine their structures and morphology.

Thick films based on the nanoparticles were prepared to detect different kinds of gases, including NH<sub>3</sub>, NO<sub>2</sub> and isoprene. The size, heat treatment and doping effects on the sensing properties were measured and discussed.

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